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# THE TENSILE DUCTILITY-TRANSITION IN MOLYBDENUM

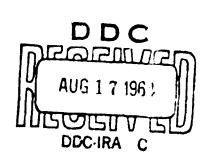
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Air Force Materials Laboratory
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# **FOREWORD**

This report was prepared by ManLabs, Inc. under USAF Contract No. AF33(657)-8424. This contract was initiated under Project No. 7351 "Metallic Materials", Task No. 735101 "Refractory Metals". The work was administered under the AF Materials Laboratory, Metals and Ceramics Division with Mr. K. Elbaum acting as project engineer.

The period covered by this report is 30 April 1963 to 30 April 1964.

#### ABSTRACT

An investigation was carried out to determine the nature of the tensile ductility transition in recrystallized molybdenum strip of three interstitial contents. For a fine grain size, the tensile ductility transition temperature (Td) and the brittleness transition temperature (Th) were found to approximately coincide; whereas with increase in grain size Td is raised and Tb is lowered. The occurrence of a minimum in the fracture stress (OF) at Td appears to be associated with the temperature dependence of the necking stress (On). Prestraining above Td, was found to result in as much as a 20% increase in  $\sigma_{\mathbf{F}}$  as determined at temperature below Td. In general, the mode of fracture initiation was found to be intergranular at Td or below and cleavage above Td. For a test temperature above as well as below Td, crack initiation appears to be the controlling step in fracture. Considering OF as a flow stress corresponding to the fracture strain (c<sub>F</sub>), the variation of O<sub>F</sub> with temperature above T<sub>d</sub> was predicted within about 15%. Based on a modification of the Cottrell relation for crack initiation, the increase in OF above Td is associated with a decrease in both the flow locking parameter  $(k_{\mathrm{f}})$  and the effective grain size.

This technical documentary report has been reviewed and is approved.

I. Perlmutter

Chief, Physical Metallurgy Branch Metals and Ceramics Division AF Materials Laboratory

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#### I. INTRODUCTION

#### A. Background

During the period of 1 November 1959 to 30 April 1963, Man Labs Inc. acted as prime contractor on a program entitled "Substructure and Mechanical Properties of Refractory Metals" under Contracts No. AF33(616)-6383 and AF33(657)-8424. Other research participants on this program were Massachusetts Institute of Technology, Rutgers the State University, University of Liverpool (England) and University of Cambridge (England). The results obtained were presented in three summary technical reports by Lement et al. (1, 2, 3)

In connection with the above program, ManLabs concentrated on the relation of microstructure to the ductile-brittle transition in refractory metals. The results obtained on molybdenum that are considered pertinent to the present investigation are as follows:

- a) For recrystallized as well as worked molybdenum strip of relatively high purity, the effective surface energy ( $\gamma$ ) at the tensile ductility-transition temperature ( $T_d$ ) as calculated on the basis of the Cottrell fracture relation is about 3000 ergs/cm<sup>2</sup>; and the corresponding critical crack length as calculated on the basis of the Griffith-Orowan fracture relation is equal to the first-order subgrain size.
- b) The discontinuous change in both ductility and fracture stress that occurs at  $T_d$  does not appear to correlate with changes in fracture mode, mechanical twinning, or the occurrence of nil-ductility. Rather the tensile ductility-transition seems to be related to the phenomenon of necking which occurs above but not below  $T_d$ . Accordingly, a suggested criterion for the occurrence of a tensile ductility transition is the intersection of the fracture stress  $(\mathfrak{I}_F)$  corresponding to uniform strain and the necking stress  $(\mathfrak{I}_n)$  vs. test temperature curves, i.e.  $\mathfrak{I}_F = \mathfrak{I}_n$  at  $T_d$ .
- c) The concept of a strain-dependent (temperature-independent) critical fracture stress was used in an attempt to predict the grain size dependence of the observed fracture stress and fracture strain based on the variation of the quantities with test temperature as measured for a fine grain, recrystallized molybdenum. The predicted values of fracture stress and fracture strain for a selected test temperature of -40°C were both found to be appreciably lower than the corresponding values actually measured as a function of grain size. This raised the question as to whether a Cottrell-type fracture relation actually holds.

#### B. Scope of Investigation

The present investigation represents a continuation of the research conducted under Contract AF33(657)-8424. The objective was to elucidate both theoretically and experimentally some of the basic factors affecting

<sup>\*</sup> Underscored numbers in parentheses designate References given at end of report.

<sup>\*\*</sup> Manuscript released by the authors May, 1964 for publication as an R&D Technical Documentary Report.

fracture phenomena in molybdenum. The approach used was to study the effects of interstitial content, grain size, and prestraining on the tensile ductility-transition behavior. Attempts were made to determine the effects of plastic strain on both flow stress and fracture stress. Comparison were made between effective crack propagation energy values as determined by tensile tests and by precracked Charpy slow bend tests.

#### II. EXPERIMENTAL PROCEDURES

#### A. Materials

The interstitial contents of the four 30-mil thick molybdenum strip materials used in this investigation are listed in Table 1. The strip from ingot Mo-E2 is relatively high in carbon (about 60-ppm); from ingot Mo-E3 is relatively high in nitrogen (about 10 ppm) and possibly oxygen (about 20 ppm); and from ingot Mo-E4 is relatively high in oxygen (about 145 ppm).

# B. Recrystallization-Anneals

All recrystallization runs were carried out in a purified argon atmosphere passing through a vertical Super-Kanthal element furnace controlled within ±8°C. Specimens were wrapped in tantalum foil and suspended vertically in the heating zone of the furnace operating at the selected recrystallization temperature. After either 0.5 or 1.0 hour at temperature (allowing for heating-up time), the specimens were removed from the heating zone and then allowed to cool to room temperature in the atmosphere. Recrystallization temperatures and times for Mo-E2, Mo-E3, and Mo-E4 are given in Table 2.

#### C. Uniaxial Prestrain

In order to strain recrystallized Mo-E2 strips and thereby attain larger grain sizes on subsequent recrystallization anneal, uniaxial prestrains in tension were utilized. Flat tensile specimens 1 inch wide and 10 inches long were machined from the molybdenum strips with the specimen axis parallel to the rolling direction. These specimens were stress-relieved for 1 hr. at 800°C in a vacuum furnace and then prestrained in uniaxial tension to about 10-15% elongation in a Baldwin tensile machine. Subsequent recrystallizations were carried out at 1300°C (medium grain size) and 1600°C (coarse grain size).

#### D. Mechanical Testing

#### 1. Tensile Tests

In general, the tensile tests were carried out in the range of  $\pm 25^{\circ}$ C to  $\pm 190^{\circ}$ C as described in the second summary technical report(2). The applied strain rate was maintained at 2.8 x  $\pm 10^{-4}$  sec  $\pm 10^{\circ}$ . In analyzing the stress-strain curves, relatively large discrepancies were found between the measured elastic modulus and the 1961 Metals Handbook value for molybdenum ( $\pm 10^{\circ}$  psi) which appears to be relatively constant in the test temperature range of 25°C to at least  $\pm 10^{\circ}$  C. In order to correct for these discrepancies, it was decided to algebraically add the difference in elastic strain (as calculated by dividing the measured stress level by the known elastic modulus and by the measured elastic modulus) to the measured strain value (elastic plus plastic).

Table 1
Chemical Composition

Ingot No.	Strip No.	Initial Condition	C ppm	N ppm	O ppm	Others
Mo-E2	Н	recrystallized at 1200°C and rolled 87% at 1600 to 400°C	65	4	12	*
	D	recrystallized at 1200°C and rolled 44% at 1250 to 900°C	65	2	11	*
	F	recrystallized at 1200°C and rolled 52% at 1250 to 400°C	56	3	8	ste se
Mo-E3	н	recrystallized at 1200°C and rolled 88% at 1000 to 400°C	19	7-15	12-29	
Mo-E4	С	rolled 64% at 1250°C and rolled 28% at 900 to 600°C	12	3	145	

\*Other elements in ppm are as follows:

<u>A1</u>	Cr	Cu	Fe	Mg	Mn	Ni	Pb	Si	Sn	Ti	v
< 50	< 5	1	30	<1	<1	< 10	< 100	10	< 50	< 10	< 10

Table 2

Procedure Used for the Control of Grain Size

Average Grain Diameter	mm.	0.020	0.00	0.310	0.023	0.020
Secondary Recrystallization Treatment	1 1 5	i 1 1	1300°C.1 hr.	1600°C,1 hr.	i i i	; ;
Uniaxial Prestrain	1 1 1	2 2 2	15%	12%	;	: :
Primary Recrystallization Treatment	1200°C,0.5 hr.	1300°C, 1 hr.	1200°C, 1 hr.	1400°C, 1 hr.	1200°C,0,5 hr.	1400°C,0.5hr.
Amount of As-Rolled Deformation	87%	44%	52%	52%	88%	28%
Strip No.	I	Ω	ы	ഥ	H	U
Ingot No.	Mo-E2				Mo-E3	Mo- E4

# E. Precracked Charpy Slow Bend Tests

Precracked Charpy slow bend tests at -100°C were carried out on specimens about 0.030 inch thick, 0.394 inch wide and 2 inches long containing a 45° V-notch. Fatigue precracking to produce an initial crack about 0.025 inch long at the V-notch was accomplished by bending in compression using a ManLabs precracking machine with the specimen in the reverse position. The slow bend tests were carried out using three point loading to determine load vs deflection characteristics.

# F. Determination of Grain Size

Grain size measurements were carried out on both longitudinal and transverse sections of the recrystallized molybianum strips. Aftermounting, the specimens were electropolished in a solution of 75 cc methyl alcohol and 25 cc H<sub>2</sub>SO<sub>4</sub> at a current density of about 1 amp./cm<sup>2</sup>. Etching was carried out using Murakami's reagent. Grain diameter measurements were made using the linear intercept method on photomicrographs taken at 100X.

#### G. Fractography

Fractured surface of tensile and slow bend test specimens were examined by both light and electron fractography. Light microscopic examination at magnifications up to 800X was used to determine where fracture initiated, mode of fracture, and facet size. Electron microscopy was carried out at 2000X on shadowed carbon replicas of the fractured surface as obtained by a) stripping a parlodion replica from the fractured surface, b) shadowing with chromium, c) depositing a carbon film by evaporation at 90, and d) dissolving the parlodion in butyl acetate.

#### III. RESULTS AND DISCUSSION

## A. Flow and Fracture Characteristics of Mo-E2 Strip

#### 1. Tensile Properties vs. Test Temperature

Plots of yield stress  $(\mathcal{O}_{Y})$ , fracture stress  $(\mathcal{O}_{F})$  and fracture strain  $(\epsilon_{F})$  for Mo-E2 molybdenum strip of fine, intermediate and coarse grain size (1) are shown in Figs. 1, 2 and 3. Discontinuous yielding was found to occur in both the fine and intermediate grain size materials and  $\mathcal{O}_{Y}$  represents the lower yield stress. On the other hand, discontinuous yielding was not observed in the coarse grain material and it was decided to use the proportional limit for  $\mathcal{O}_{Y}$ . This seems justified on the basis that the proportional limits of the fine and coarse grain materials were found to be approximately equal to their respective lower yield stress values.

Based on Figs. 1, 2 and 3, the brittleness-transition  $(T_b)$ , ductility-transition  $(T_d)$ , and fracture stress maximum  $(T_m)$  temperatures\* as well as both the corresponding fracture strains  $(\epsilon_F)$  and ratios of  $\sigma_F$  to  $\sigma_V$  at  $\sigma_D$  are given in Table 3. The values obtained for the fine and intermediate grain sizes of the Mo-E2 strip are about the same except that  $\sigma_D$  is lower for the intermediate grain size. For the fine grain Mo-E2, it was found that  $\sigma_D$  approximately coincides with  $\sigma_D$ . The coarse grain size has the lowest  $\sigma_D$  and the highest  $\sigma_D$  and  $\sigma_D$  temperatures. Also, the values of  $\sigma_D$  and  $\sigma_D$  are highest for the coarse grain size.

#### 2. Dependence of Yield Stress on Grain Size

Based on Figs. 1, 2 and 3, plots of  $\sigma_y$  vs  $t^{-1/2}$  are shown in Fig. 4 for the Mo-E2 strip subjected to test temperatures in the range of +25 to -150°C. The corresponding  $k_y$  values (based on normal stress and full grain size) are given by the slopes of these plots. As shown in Fig. 5,  $k_y$  appears to increase linearly with decrease in test temperature, and the extrapolated value at -196°C is about six times higher than at +25°C. The  $k_y$  ratio at -80°C is about three, which is about twice that reported for molybdenum by Wronski (4) (about 1.6). This is tentatively attributed to the fact that the interstitial level of the molybdenum (Mo-E2) used in the present investigation is significantly lower than that used by Wronski.

# 3. Prediction of Dependence of Fracture Stress on Grain Size

In the previous report (3), a method of predicting the variation of observed fracture stress with grain size over the test temperature range of  $T_d$  to  $T_m$  based on data for a single grain size was presented. It was assumed that the critical fracture stress ( $O_c$ ) is strain-dependent but temperature-independent, and that the Cottrell (5) relation is obeyed if  $O_c$  at given fracture strain ( $E_r$ ) is determined as a function of the grain size (f):

$$\left[\sigma_{c}\right]_{\epsilon_{F}} = C_{\epsilon}I^{-1/2} \tag{1}$$

<sup>\*</sup>These temperatures are defined in Appendix I.

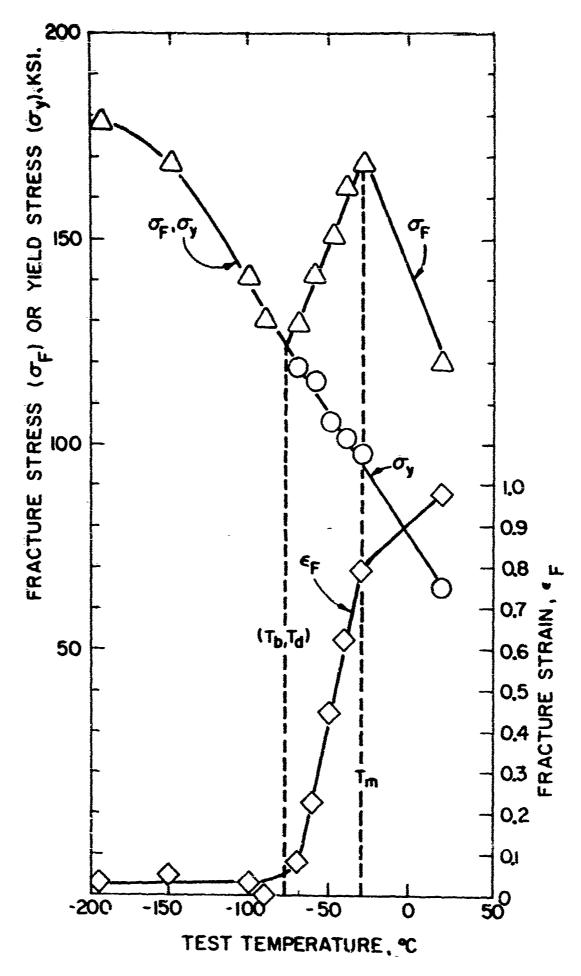


Fig. 1 - Effect of Test Temperature on the Tensile Properties of Recrystallized Molybdenum (Mo-E2) Strip (I = 0.026 mm).

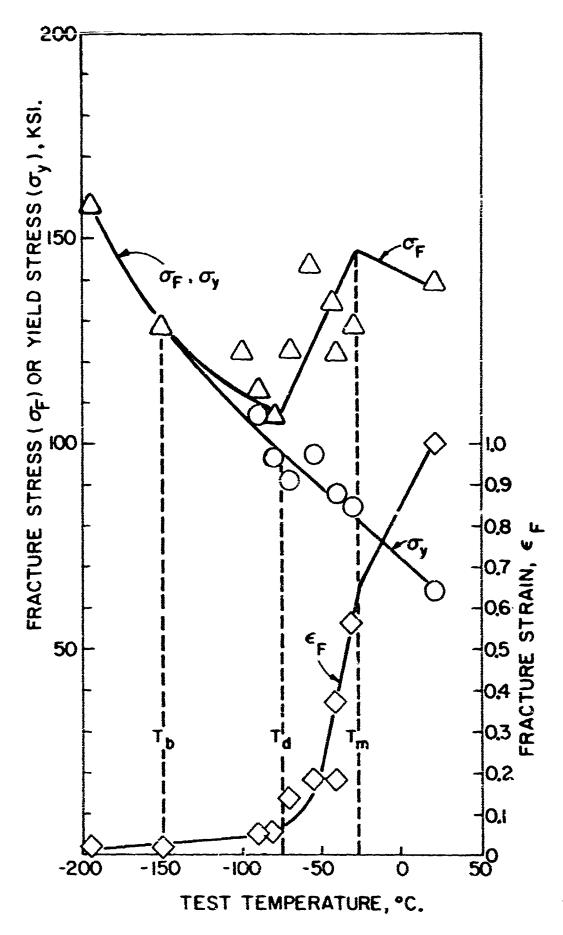


Fig. 2 - Effect of Test Temperature on Tensile Properties of Recrystallized Molybdenum (Mo-E2) Strip (1 = 0.044 mm).

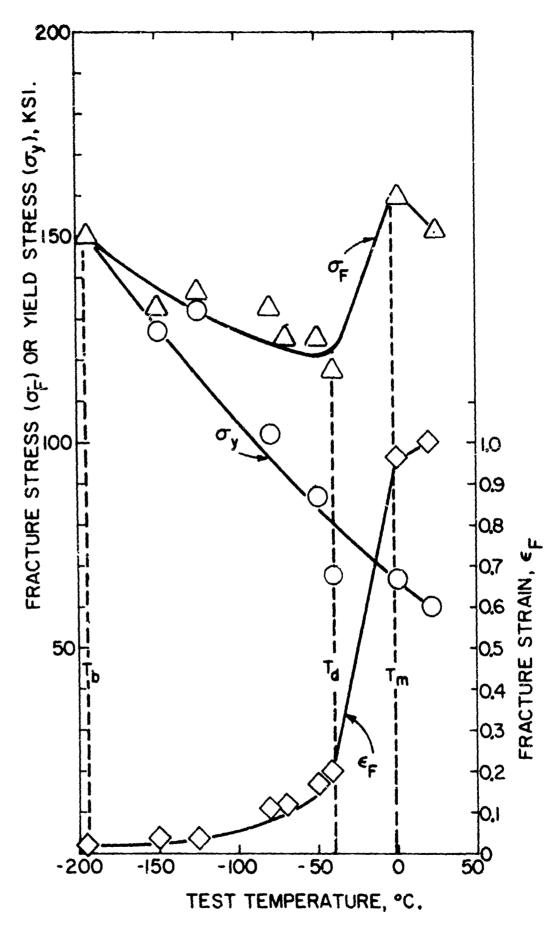


Fig. 3 - Effect of Test Temperature on Tensile Properties of Recrystallized Molybdenum (Mo-E2) Strip (l = 0.174 mm).

Table 3

Tensile Test Characteristics of Mo-E2, Mo-E3 and Mo-E4 Strip

<u>Strip</u>	Average GrainSize mm	$\frac{T_b}{^{\circ}C}$	F at T,	$\frac{T_d}{^{\circ}C}$	F at Td	T <sub>m</sub>	F at T m	σ <sub>F</sub> /σ <sub>y</sub> at T <sub>d</sub>
Mo-E2	0.026	-80	0.04	-80	0.04	-30	0.80	1.00
	0.044	-150	0.02	-75	0.07	-25	0.65	1.10
	0.174	-195	0.02	-40	0.18	0	0.95	1.44
Mo-E3	0.023	-80	0.03	-80	0.03	-20	0.90	1.00
Mo-E4	0.020	-35	0.00	-35	0.00	+25	0.62	1.00

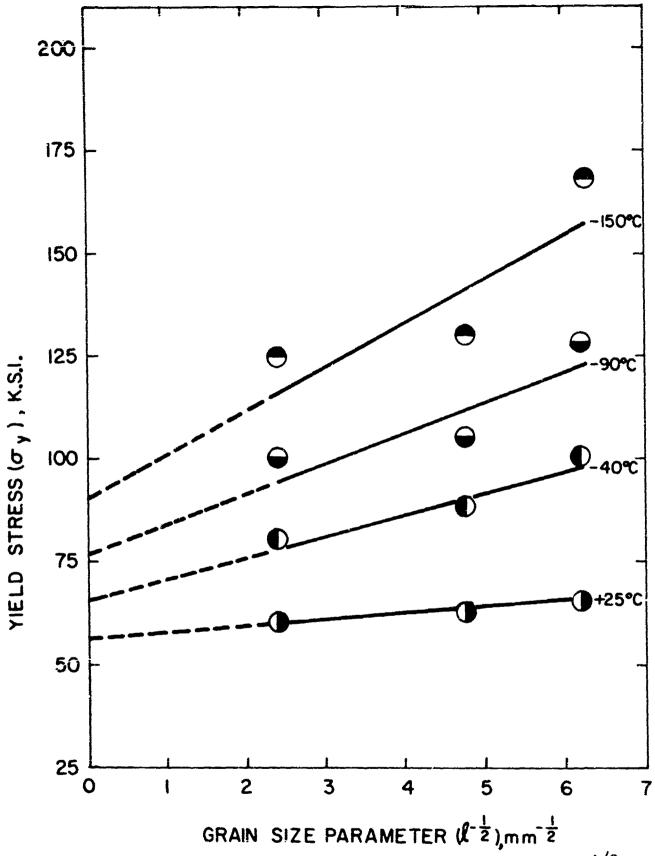


Fig. 4 - Variation of Yield Stress (σ<sub>y</sub>) with Grain Size (f<sup>-1/2</sup>) of Molybdenum (Mo-E2) Strip in Test Temperature Range of +25 to -150°C.

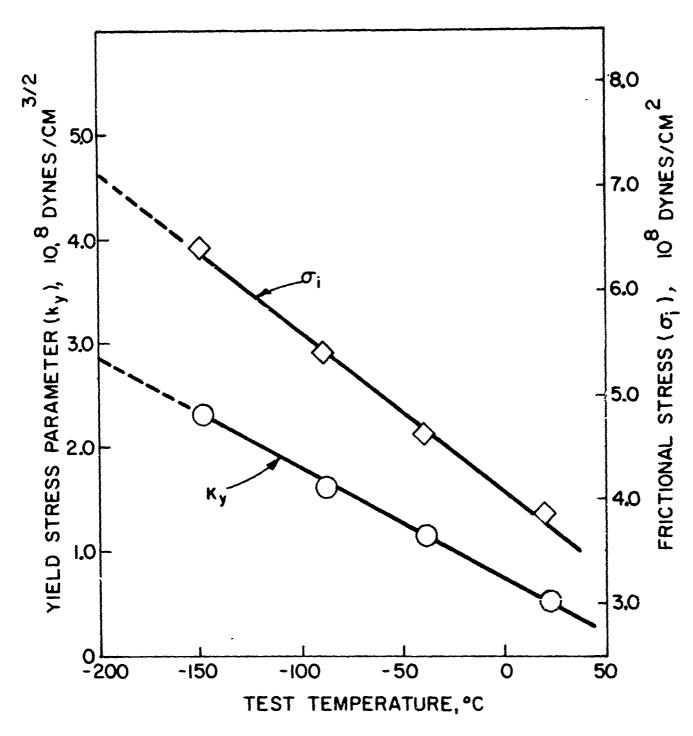


Fig. 5 - Variation of Yield Stress Parameter (ky) and Frictional Stress (C<sub>i</sub>) of Molybdenum (Mo-E2) Strip with Test Temperature. The Parameter K and Frictional Stress of are Based on Normal Stress (Vy) and Full Grain Diameter (f).

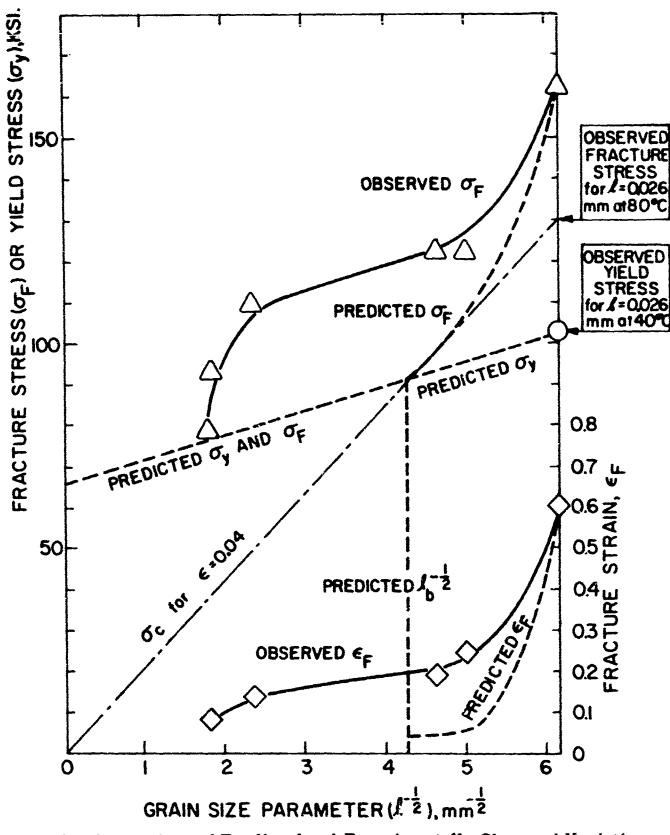


Fig. 6 - Comparison of Predicted and Experimentally Observed Variations of the Fracture Stress (O<sub>F</sub>) and Fracture Strain (e<sub>F</sub>) with Grain Size (l<sup>-1</sup>/2) of Recrystallized Molybdenum (Mo-E2) Strip for a Test Temperature of -40°C Based on Data Obtained for Fine Grain Size (l = 0.026 mm).

Fig. 6 is a revised plot showing both the observed and predicted values of the fracture stress ( $O_F$ ) and fracture strain ( $e_F$ ) as function of  $I^{-1}/2$ . The observed fracture stresses are as much as 30% higher than predicted; and the observed fracture strain are as much as four times the predicted values. This raised the question as to whether the critical fracture stress actually follows the relation given in Eq.(1). In order to clarify this question, plots of  $O_F$  vs  $I^{-1/2}$  corresponding to  $e_F$  values of 0.2, 0.4 and 0.6 were made based on Figs. 1, 2 and 3. As shown in Fig. 7, the slopes ( $e_F$ ) are all about 0.9 x  $e_F$  108 cgs, and there is an intercept ( $e_F$ ) corresponding to infinite grain size. It appears that the dependence of  $e_F$  at constant fracture strain has the following form:

$$[\sigma_{\mathbf{F}}]_{\epsilon} = \sigma_{\mathbf{F}_{\infty}} + k_{\mathbf{F}} \ell^{-1/2}$$
 (2)

Eq. (2) differs from the form of the Cottrell (5) fracture relation, Eq.(1), according to which  $\sigma_F$  or  $\sigma_c$  should equal zero at infinite grain size. Thus, it does not seem possible to accurately predict the dependence of  $\sigma_F$  on test temperature based on data obtained for a single grain size, as was attempted by this approach.

#### 4. Prediction of Fracture Stress from Fracture Strain

#### 4.1 Previous Approach

In the previous report (3), calculations were made of the ratio of the maximum to minimum fracture stress in the transition range for Mo-El strip (about 40 ppm carbon, 4 ppm nitrogen and 25 ppm oxygen) rolled 5, 46 and 88% (reduction in area) after recrystallization; and for recrystallized Mo-E2 strip in the fine grain condition. For the Mo-El strips,  $T_d$  and  $T_m$  appear to coincide; whereas for the Mo-E2 strip (Fig. 1)  $T_m$  is about 50°C higher than  $T_d$ . It was assumed that the over-all strengthening factor due to necking (qn) is equal to the following product:

$$q_n = q_i \times q_p \times q_i = (0_F)_{max}/(0_F)_{min}$$
 $q_p = plastic constraint factor$ 
 $q_i = strain rate factor$ 

(3)

Calculated values of  $q_n$  based on Eq. (3) were found to agree within 10% of the corresponding measured values.

Subsequent analysis of this approach has led to the conclusion that the good agreement obtained between calculated and measured values of  $q_n$  is probably fortuitous. This is based on the following considerations:

a) The strengthening factor associated with substructural

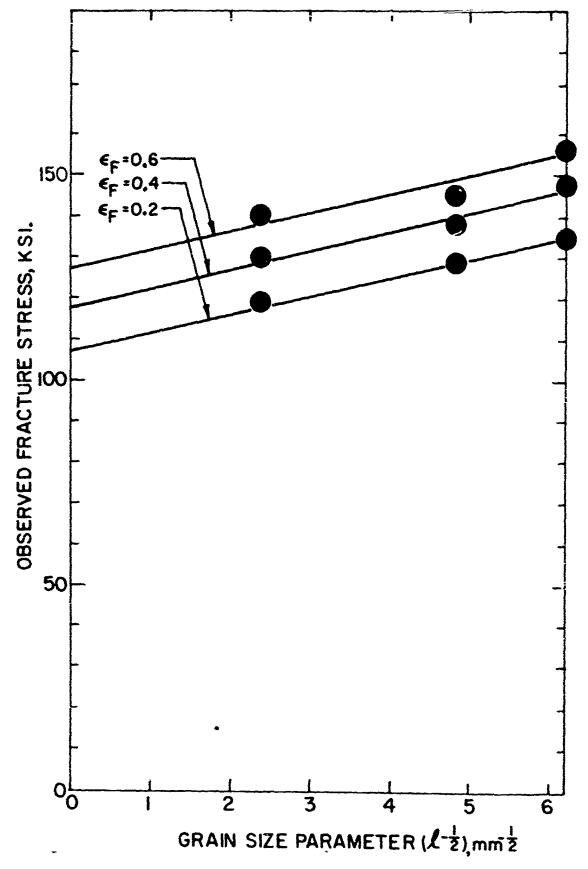


Fig. 7 - Variation of the Observed Fracture Stress (O<sub>F</sub>) at Constant Strain with Grain Size (f<sup>-1</sup>/2) for Recrystallized Molybdenum Strip (Mo-E2).

changes during straining from the necking stress to the fracture stress,  $(\sigma_F)_{\max}$ , at  $T_{\min}$  was not taken into account.

b) The calculated grain size factor  $(c_i)$  is probably too large since the fracture stress (which is the flow stress at fracture strain) was assumed to be directly proportional to  $i^{-1/2}$  whereas the actual dependence of flow stress includes a term representing the flow stress at infinite grain size.

c) The strengthening factors are more likely to be additive rather than multiplicative.

#### 4.2 Revised Approach

Based on the foregoing considerations, it was decided to modify Eq. (3) and attempt to predict the variation of fracture stress with test temperature above  $T_d$ . Since the fracture stress and the flow stress at the fracture strain are identical, the factors that influence flow stress can be utilized to calculate the fracture stress provided that the fracture strain is known. Assuming that the observed flow stress  $O_f$  is equal to the flow stress due to substructural changes,  $O_{fs}$ , plus the strengthening contributions of a) decreased grain size during straining, b) plastic constraint due to necking, and c) increased strain rate due to deformation being confined to the neck; the following relation should hold:

$$\sigma_{f} = \sigma_{fs} \left[ 1 + (q_{f} - 1) + (q_{p} - 1) + (q_{e} - 1) \right]$$
 (4)

where  $q_f$ ,  $q_p$  and  $q_e$  are the strengthening factors due to grain size, plastic constraint and strain rate (in the necked region) respectively, corresponding to a given amount of strain  $\epsilon$  (elastic plus plastic). As is subsequently discussed,  $q_f = 1$  and therefore can be neglected. For the prediction of fracture stress ( $O_F$ ), the identity of  $O_F$  and  $O_f$  at  $\epsilon_F$  is utilized and equation (4) takes the following form:

$$\sigma_{F} = \sigma_{Fs} [1 + (q_{p} - 1) + (q_{\epsilon} - 1)]$$
 (5)

where  $\mathfrak{C}_{Fs}$  is the contribution of substructural changes to the fracture stress, and  $\mathfrak{q}_p$  and  $\mathfrak{q}_\epsilon$  are maximum values which occur at the fracture strain  $(\mathfrak{c}_F)$ .

#### (a) Substructural Strengthening

For the determination of  $\sigma_{F_S}$  it is necessary to know the relation of the true flow stress ( $G_{F_S}$ ) to the true strain ( $\epsilon$ ). The simplest

assumption is that the so called power law holds:

$$\sigma_{fs} = K(\epsilon)^n$$
 (6)

where  $\epsilon$  = total strain (elastic plus plastic)

K = strain hardening coefficient
n = strain hardening exponent

In particular,  $0_{Fs}$  is given by substituting  $\epsilon_F$  for  $\epsilon$ :

$$\sigma_{Fs} = K(\epsilon_F)^n \tag{7}$$

Thus it is possible to determine  $\mathcal{O}_{FS}$  provided K, n and  $\epsilon_F$  are known. The best way to determine K and n is from a plot of Eq. (6) in the form of log  $\mathcal{O}_{fs}$  vs log  $\epsilon$  using data corresponding to the partion of the stress-strain curve from the yield stress  $(\mathcal{O}_{v})$  to the necking stress  $(\mathcal{O}_{n})$ .  $\mathcal{O}_{Fs}$  can then be determined using equation (7), which is effectively extrapolating the log  $\mathcal{O}_{fs}$  vs log  $\epsilon$  plot to log  $\epsilon_{F}$ . This assumes that the power law continues to hold from  $\mathcal{O}_{v}$  to  $\mathcal{O}_{F}$  (or from the strain  $(\epsilon_{v})$  at  $\mathcal{O}_{v}$  to  $\epsilon_{F}$ ).

An alternative way to determine  $\sigma_{Fs}$  is to utilize the relation between the yield stress ( $\sigma_y$ ) and the yield strain ( $\epsilon_y$ ):

$$\sigma_{y} = K \left( \epsilon_{y} \right)^{n} \tag{8}$$

Defining  $q_s$  as  $\sigma_{Fs}/\sigma_y$  at the fracture strain ( $\epsilon_F$ ) and utilizing Eq. (7) and (8) gives

$$q_{s} = \left(\frac{\epsilon_{F}}{\epsilon_{y}}\right)^{n} \tag{9}$$

Therefore, q represents the substructural strengthening factor and  $\sigma_{Fs}$  can be determined if n,  $\epsilon_{V}$  and  $\epsilon_{F}$  are known.

# (b) Grain Size Strengthening Factor

The grain size factor (q) may be defined as follows:

$$q_{i} = \frac{\sigma_{fs} + \Delta\sigma_{fs}}{\sigma_{fs}}$$
 (10)

where  $\Delta \sigma_{fs}$  = increase in the flow stress due to the decrease in transverse grain dimensions. The dependence of flow stress  $\sigma_{fs}$  at a given strain and test temperature on grain diameter is given by the following relation:

$$\sigma_{fs} = \sigma_{fs} + k_f t^{-1/2} \tag{11}$$

where  $\sigma_{f_{\infty}}$  = flow stress at infinite grain size  $k_f$  = flow stress parameter

Since a decrease in grain size occurs because of the reduction in area during the tensile test, the increase in flow stress is at a given strain is given by

$$\Delta \sigma_{fs} = k_f \Delta (f^{-1/2}) \tag{12}$$

and therefore

$$q_{\ell} = 1 + \frac{k_{f} \Delta(\ell^{-1/2})}{\sigma_{fs}}$$
 (13)

Assuming that  $k_f = 0.3$  k, for molybdenum and using the measured value of  $k_c$  at  $20^{\circ}$ C for Mo-E2 (1.8 ksi/mm<sup>-1/2</sup>), the corresponding value of  $k_f$  at  $20^{\circ}$ C is 0.6 ksi/mm<sup>-1/2</sup>. For a reduction of area as high as 69% ( $\epsilon = 1.00$ ),  $\Delta(f^{-1/2}) = 2$ mm<sup>-1/2</sup>. Since the approximate value of  $t_f$  at  $t_f$  at  $t_f$  calculated on the basis of Eq. (13) is about 1.01. Since this represents a maximum value,  $t_f$  can in most cases be considered as equal to unity.

# (c) Plastic Constraint Strengthening Factor

Based on the Bridgeman (6) correction, the plastic constraint factor (q), for a round tensile specimen is approximately given by the following relation in terms of the fracture strain  $(\epsilon_F)_{rd}$ .

$$(q_p)_{rd} - 1 = \frac{0.15 (\epsilon_F)_{rd}}{1 - 0.15 (\epsilon_F)_{rd}}$$
 (14)

According to an analysis of Aronofsky's (7) results for a flat specimen, the following relations appear to hold between the plastic constraint factors,  $(q_p)_{fl}$  and  $(q_p)_{rd}$  and between the fracture strains,  $(\epsilon_F)_{fl}$  and  $(\epsilon_F)_{rd}$  for

a flat and round specimen, respectively:

$$(q_p)_{fl} - 1 = 0.5 [(q_p)_{rd} - 1]$$
 (15)

$$(\epsilon_F)_{ff} = 0.9 (\epsilon_F)_{rd}$$
 (16)

Solving Eq. (14), (15) and (16) for  $(-1)_{ff}$  in terms of  $(\epsilon_F)_{ff}$  and dropping the subscript ff gives

$$q_{p} = \frac{0.5}{0.5 - 0.08 \, \epsilon_{F}} \tag{17}$$

# (d) Strain Rate Strengthening Factor

As discussed in the previous report (3) the strain rate (i) in the necked region of a tensile specimen is higher than the applied strain rate and the flow stress required to produce a given strain is increased by a factor(q:) given by the following equation:

$$q_{\xi} = \left[ \frac{L_{g(\epsilon_{n} - \epsilon_{n})^{1/4}}}{\left[ 8 a_{o}^{a_{u}} \exp^{-\epsilon_{n} - 8 a_{o}^{2}} \exp^{-2\epsilon_{n} - 4 (\epsilon_{n} - \epsilon_{u})^{1/2} (a_{u} - a_{o}^{e} \exp^{-\epsilon_{n})^{2}} \right]^{1/2}} \right]^{r}$$
(18)

where  $L_g = \text{gage length}$   $\epsilon_n$  and  $\epsilon_u = \text{total necking strain and maximum uniform strain respectively}$ 

a and a = initial specimen semi-width and specimen semi-width corresponding to maximum uniform strain respectively

> r = strain rate exponent (0.05 for molybdenum according to Bechtold's (8) results).

Based on the previous calculations (3), the factor  $q_{\epsilon}$  has the following dependence on plastic strain as given by the difference between  $\epsilon_F$  and the maximum uniform strain ( ; ):

F-tu	<u>•</u> •
0.0001	1.03
0.0010	1.06
0.0100	1.09
0.1000	1.11
0.5000	1.11

Since the values of  $\epsilon_F - \epsilon_u$  fall in the range of about 0.10 to 0.8 for the Mo-E2 strip tensile test results considered, it may be assumed that  $q_{\epsilon}$  is equal to 1.11 for all the cases of interest.

# 4.3 Calculation of Fracture Stress Assuming Completely

#### Uniform Elongation

As previously defined,  $\sigma_{FS}$  represents the fracture stress due to substructural changes alone, i.e. the flow stress corresponding to the observed fracture strain assuming completely uniform elongation (no necking). In order to calculate  $\sigma_{FS}$  for a given test temperature, it is necessary to first determine the strain hardening exponent (n) and strain hardening coefficient (K) from a plot of  $\log \sigma_{fS}$  vs  $\log \epsilon$  at that temperature and then to utilize the power law as given in Eq. (8).

The variations of n and K with test temperature in the range from  $T_d$  to 20°C are shown in Figs. 8\* to 10 for Mo-E2 (fine, intermediate and coarse grain sizes). Corresponding sets of n, K and  $\epsilon_F$  values were used to calculate  $0_{Fs}$  at  $T_d$ , at half-way between  $T_d$  and  $T_m$ , at  $T_m$  and at 25°C. The results given in Table 4 indicate that  $0_{Fs}$  goes through a maximum with increase in test temperature above  $T_d$  just as does the observed fracture stress. However, the calculated maximum in  $0_{Fs}$  occurs at about half-way between  $T_d$  and  $T_m$  instead of at  $T_m$  as does the maximum in the observed fracture stress.

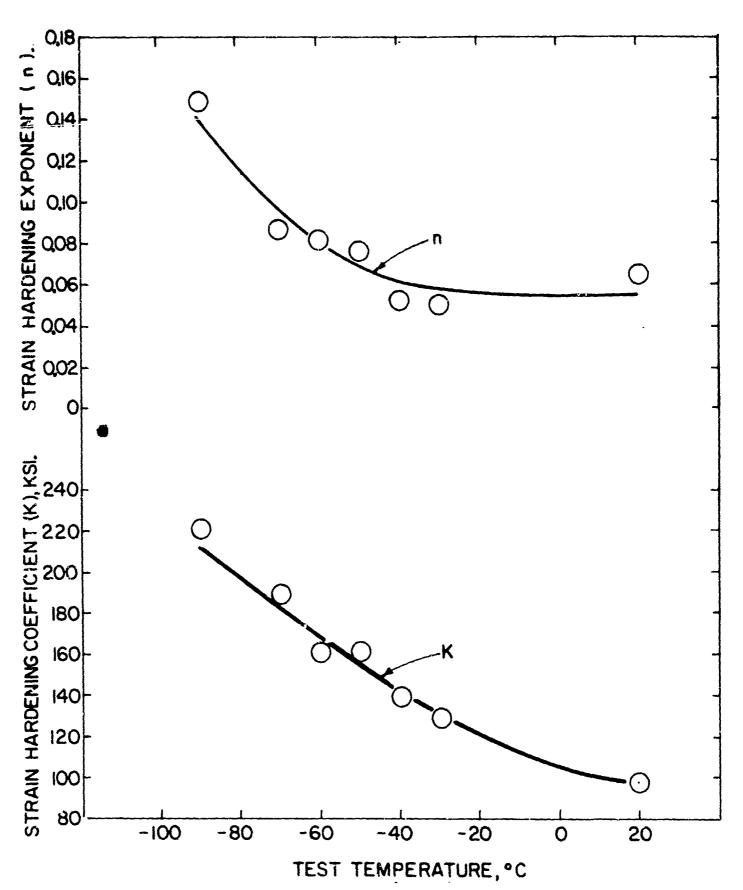
From the calculated values of  $\sigma_{Fs}$  and corresponding values of observed yield stress  $(\sigma_v)$ , it is possible to calculate the substructural strengthening factor  $(q_s)$  which was defined as  $\sigma_{Fs}/\sigma_v$ . As shown in Table 4, the variation of  $q_s$  with increase in test temperature above  $T_d$  consists of a steady increase up to at least 25°C for the fine grain Mo-E2 strip; whereas  $q_s$  reaches a maximum and then decreases for the intermediate and coarse grain Mo-E2 strip.

# 4.4 Calculation of Fracture Stress Under Actual Necking

#### Conditions

Based on the revised approach, the relation between the fracture stress under actual necking conditions  $(0_F)$  and the fracture stress due

<sup>\*</sup>The values given in Fig. 8 for n and K at -90°C (i.e., below  $T_d = T_b = -85$ °C) correspond to a specimen that actually necked at -90°C.



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Fig. 8 - Variation of Strain Hardening Exponent (n) and Strain Hardening Coefficient (K) with Test Temperature for Recrystallized Molybdent (Mo-E2) Strip (1 = 0.026 mm).

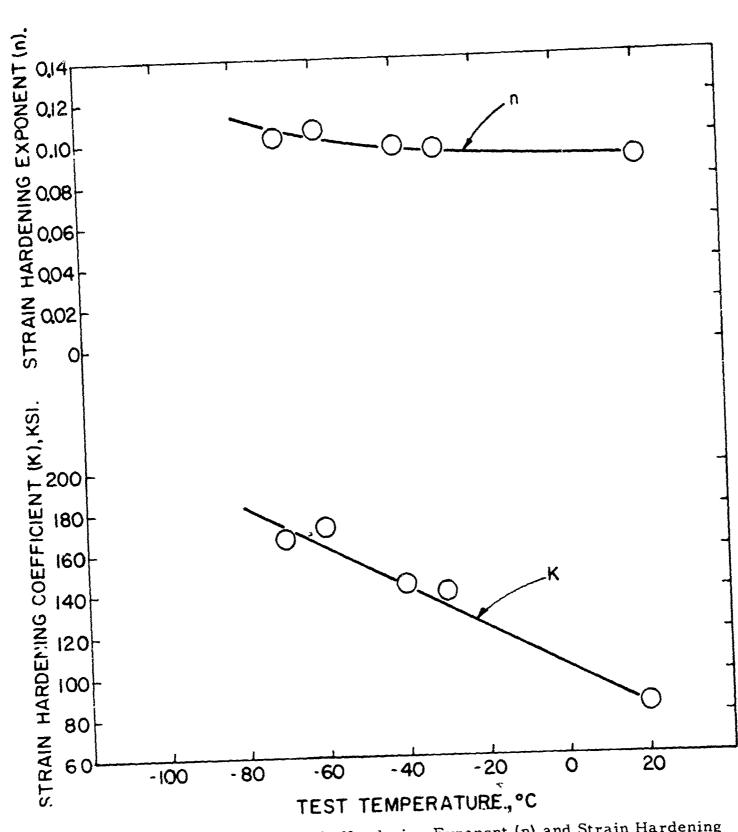
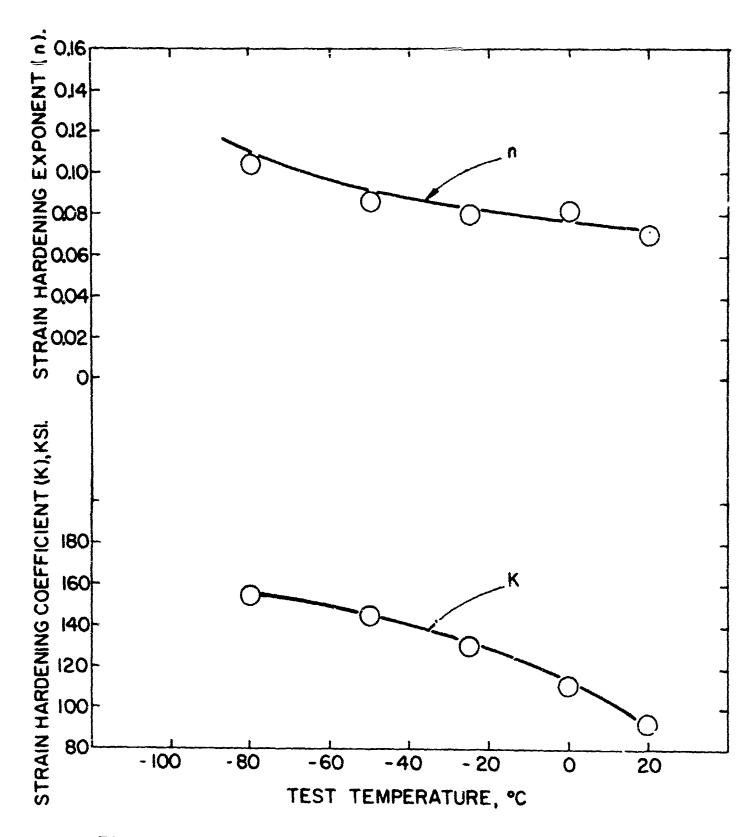


Fig. 9 - Variation of Strain Hardening Exponent (n) and Strain Hardening Coefficient (K) with Test Temperature for Recrystallized Molybdenum (Mo-E2) Strip (\$\ell = 0.044\$ mm).



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Fig. 10 - Variation of Strain Hardening Exponent (n) and Strain Hardening Coefficient (K) with Test Temperature for Recrystallized Molybdenum (Mo-E2) Strip (1 = 0.174 min).

Calculated Values of Fracture Stress Assuming Completely Uniform Elongation

Substructural Strengthening Factor	1.08 1.31 1.37	1.50 1.36 1.48 1.54	1.31 1.47 1.70 1.68	1.54 1.20 1.36 1.30	1.20 1.41 1.98 2.79 3.18
Observed Yield Stress	125 112 94	65 90 81	65 73 66	60 132 109 97	62 62 48 38
Calculated Uniform Fracture Stress OFS	135 147 129	133 133 125	85 119 124 111		104 123 134 121
Strain Hardening Coefficient K	195 160 130 97	180 155 130 85	140 129 111 92	216 156 126 100	164 156 150 145
(e F) n	0.69 0.92 0.99 1.00	0.74 0.86 0.96 1.00	0.85 0.96 1.00 1.00	0.73 0.95 1.00 1.00	0.63 0.79 0.89 0.86
Strain Hardening Exponent	0.116 0.074 0.058 0.054	0.109 0.100 0.094 0.090	0.088 0.081 0.077 0.072	0.090 0.062 0.052 0.046	0.200 0.236 0.276 0.311
Fracture Strain	0.04 0.35 0.80 0.98	0.07 0.20 0.65 1.00	0.17 0.62 0.95 1.00	0.03 0.54 0.91 1.06	0.10 0.36 0.68 0.61
Test Temp	-80 -55 -30 +25	-75 -50 -25 +25	-40 -20 0 +25		-25 0 +25 +50
Material & Grain Size	(0.026mm)	Mo-E2 (0.044mm)	Mo-E2 (0. 174mm)	Mo-E3 (0.023mm)	Mo-E4 (0.020m.a)

to substructural changes alone  $(\overline{V}_{\Gamma S})$  is given by equation (5), which requires that  $q_D$  and  $q_E'$  be known as a function of  $\epsilon_F$ . Eq. (17) was used to calculate  $q_D$  at the same temperatures at which values of  $\overline{V}_{FS}$  given in Table 4 were obtained for the three grain sizes of Mo-E2 strip. Taking  $q_E'$  as approximately 1. 11 based on Eq. (18), calculations were made of  $\overline{V}_F$  as a function of test temperature. The calculated values of  $\overline{V}_F$  shown in Table 5 agree with the observed fracture stress values within about 15% for 10 cases, and within about 20% for the other two cases. This is considered to be good agreement since the precision of measurement of fracture stress is about 10%.

The calculated maxima in  $\sigma_F$  occur about half-way between  $T_d$  and  $T_m$  similar to the calculated  $\sigma_{Fs}$  maxima. However, there is less of a difference between the calculated maximum in  $\sigma_F$  and the calculated value at  $T_m$  as compared to the corresponding values of  $\sigma_{Fs}$ . It, therefore, appears that the method used to calculate  $\sigma_F$  is correct in principle but suffers from inaccuracies in determing values of  $\sigma_{Fs}$  and  $\sigma_{Fs}$ .

# B. Flow and Fracture Characteristics of Mo-E3 Strip

# 1. Tensile Properties vs. Test Temperature

The effect of test temperature on the tensile properties of Mo-E3 strip of fine grain size (0.023 mm) is shown in Fig. 11. Similar to the fine grain Mo-E2 strip, it was found that  $T_{\dot{b}}$  and  $T_{\dot{d}}$  coincide. As indicated in Table 3, the tensile test characteristics of Mo-E2 and Mo-E3 are about the same.

The variations of the strain hardening exponent (n) and the strain hardening coefficient (K) of Mo-E3 with test temperature are shown in Fig. 12. This information was utilized to calculate the variation of fracture stress ( $\sigma_F$ ) assuming completely uniform elongation (Table 4) as well as the fracture stress ( $\sigma_F$ ) under actual necking conditions (Table 5). It was found that the substructural strengthening factor ( $\sigma_F$ ) goes through a maximum at about -45°C. This is reflected in the occurrence of the calculated maximum  $\sigma_F$  value at -45°C instead of at -20°C as actually observed.

#### 2. Effect of Uniform Prestraining

Experiments were carried out on Mo-E3 to determine the effect of a uniform prestrain  $(\epsilon_{pr})$  at a temperature above  $T_d$  on the fracture stress  $(0_F)$  and fracture strain  $(\epsilon_F)$  at a temperature below  $T_d$ . The temperatures selected for the prestraining,  $-20^{\circ}$ C and  $+25^{\circ}$ C, correspond to the temperature at which  $0_F$  goes through a maximum  $(T_m)$  and room temperature respectively. The procedure was to prestrain to increasing values of  $\epsilon_{pr}$  up to about 0.19 at  $-20^{\circ}$ C or  $+25^{\circ}$ C, decrease the load by about 90%, cool to a final test temperature  $(-100^{\circ}$ C,  $-150^{\circ}$ C, or  $-196^{\circ}$ C), and load until fracture occurs.

As shown in Fig. 13, the fracture stress ( $G_F$ ) at  $-100^{\circ}$ C increases with prestrain ( $\epsilon_{pr}$ ) and goes through a maximum at  $\epsilon_{pr} = 0.1$ . The maximum in  $G_F$  corresponds to an increase of about 30% as compared to  $G_F$  for zero prestrain as measured at the same breaking temperature of  $-100^{\circ}$ C. For a breaking

Calculated Values of Fracture Stress Under Actual Necking Conditions

Ratio of Calc. to Obs.	1.08	0 270		0.79 1.13 1.10 0.90	00 mm Nm
Fracture Stress $0_{\overline{F}}$ calc. obs.	ksi 125 145 168	W 70 70 80	m 01 +# .0	m	0 0 - 0 0
Fra St	135 172 163	V 20 20	الشاماني	N 10 2 . 0 a	0 - 41,0 41
Strenghthening Factors	1.00	: 8777	- 677		
Strenghth	1.00	00-		0778	1.02 1.06 1.12 1.11
Uniform Fracture Stress OFS	135 147 129 97	133 133 125 85	119 124 111 92	158 148 126 100	104 123 134 121
Fracture Strain	0.04 0.35 0.80 0.98	0.07 0.20 0.65 1.00	0.17 0.62 0.95 1.00	0.03 0.54 0.91 1.06	0.10 0.36 0.68 0.61
Test Temp	-80 -55 -30 +25	-75 -50 -25 +25	-40 -20 -20 +25	-85 -45 -20 +25	. 25 . 0 . 4 . 50 . + 50
Material & Grain Size	Mo-E2 (0.026mm)	Mo-E2 (0.0 <b>4</b> 4mm)	Mo-E2 (0. 174mm)	Mo-E3 (0.023mm)	Mo-E4 (0.020mm)

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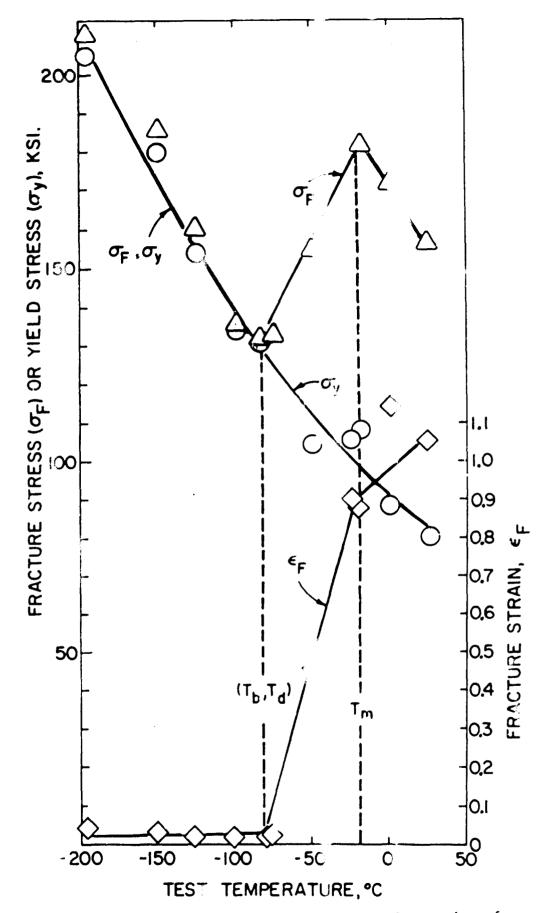


Fig. 11 - Effect of Test Temperature on Tensile Properties of Recrystallized Molybdenum (Mo-E3 St ip (f = 0.023 mm).

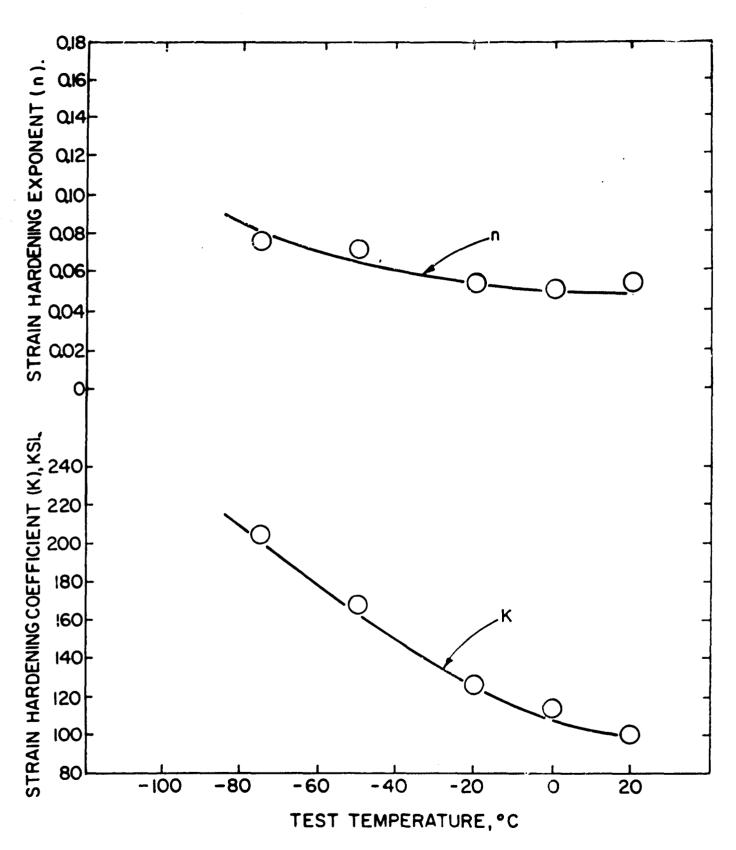


Fig. 12 - Variation of Strain Hardening Exponent (n) and Strain Hardening Coefficient (K) with Test Temperature for Recrystallized Molybdenum (Mo-E3) Strip (1 = 0.023 mm).

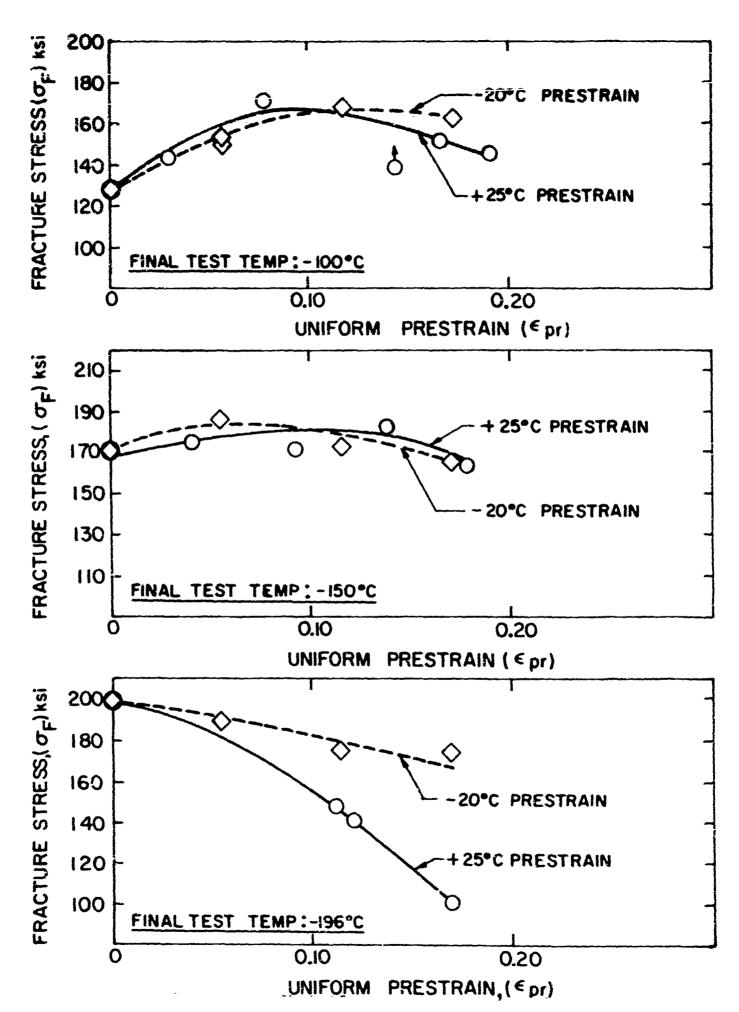


Fig. 13 - Effect of Uniform Prestraining Mo-E3 at -20°C and +25°C on the Fracture Stress at -100°C, -150°C, and -196°C.

temperature of -150°C there appears to be a slight maximum in  $\sigma_F$  at  $\varepsilon_{pr} \equiv 0.06$  for a prestrain temperature of -20°C. A similar trend was found for prestraining at +25°C although the position of the maximum is in doubt because of the scatter in the data. At -196°C,  $\sigma_F$  decreases with prestrain and the rate of decrease is greater for the +25°C than for the -20°C prestrain temperature.

The corresponding variations of the fracture strain ( $\epsilon_F$ ) and the total strain ( $\epsilon_{pr} + \epsilon_F$ ) with prestrain  $\epsilon_{pr}$  are shown in Figs. 14, 15 and 16. After prestraining at either -20 or + 25°C,  $\epsilon_F$  as a function of  $\epsilon_{pr}$  goes through a maximum at all of the final test temperatures (-100°C, -150°C, and -196°C). The total strain ( $\epsilon_{pr} + \epsilon_F$ ) increases with  $\epsilon_{pr}$  and tends to level off at the higher values of  $\epsilon_{pr}$ .

The variation of  $\sigma_F$  with total strain ( $\epsilon_{pr} + \epsilon_F$ ) is shown in Table 6 for Mo-E3 strip prestrained at -20°C and broken at -100, 150, and -196°C. It was found that  $\sigma_F$  increases appreciably at -100°C, increases slightly at -150°C, and decreases at -196°C as the total amount of strain is increased. For the purpose of comparison,  $\sigma_F$  values obtained in the range of -80 to -67°C without any prestrain are listed in Table 6. The ratio of fracture stresses with and without any prestrain varies with both total strain and test temperature. This is further evidence that  $\sigma_F$  is not a unique function of strain alone.

### 3. Effect of Necking Prestrain

Studies were carried out to determine the effect of necking prestrains at  $+25^{\circ}$ C on the fracture stress ( $\sigma_{\rm F}$ ) and fracture strain ( $\varepsilon_{\rm F}$ ) at  $-100^{\circ}$ C. As shown in Table 7, a necking prestrain of 0.29 at  $+25^{\circ}$ C results in an increase in  $\sigma_{\rm F}$  at  $-100^{\circ}$ C of about 20% and in  $\varepsilon_{\rm F}$  at  $-100^{\circ}$ C of about 500% (0.03 to 0.18). A larger necking prestrain (0.64) results in a slight increase in  $\sigma_{\rm F}$  along with a 55% decrease in  $\varepsilon_{\rm F}$  as compared to a 0.29 necking prestrain.

In order to determine to what extent the geometry of the neck affects  $\sigma_F$  and  $\varepsilon_F$ , simulated necking was studied using two procedures: a) prestraining at +25°C to produce a neck corresponding to a strain of 0.64 and recrystallizing to remove the deformation; b) machining a neck in the rolling plane section of the specimen that corresponds to 0.64 strain. As shown in Table 7 procedures a) and b) resulted in completely brittle fractures ( $\varepsilon_F = 0$ ) as well as decreases in  $\sigma_F$  of 30% and 12% respectively as compared to a non-prestrained specimen tested at -100°C ( $\sigma_F = 140$  ksi). These results indicate that the geometrical effect of a neck corresponding to 0.64 strain is to provide a stress concentration which results in a lowering of  $\sigma_F$ . It should be noted that the simulated neck obtained by procedure b) was machined in only one plane, whereas during tensile deformation necking actually occurs along both the thickness and width dimensions. Therefore, a greater stress concentration is presumably present in a specimen that is pre-necked by tensile deformation, procedure a), than by machining, procedure b).

In view of the simulated necking results, it appears that the plastic deformation associated with prestraining has a greater effect on raising Opeat -100°C than is indicated by the 30% increase shown in Table 7 for 0,64 necking prestrain.

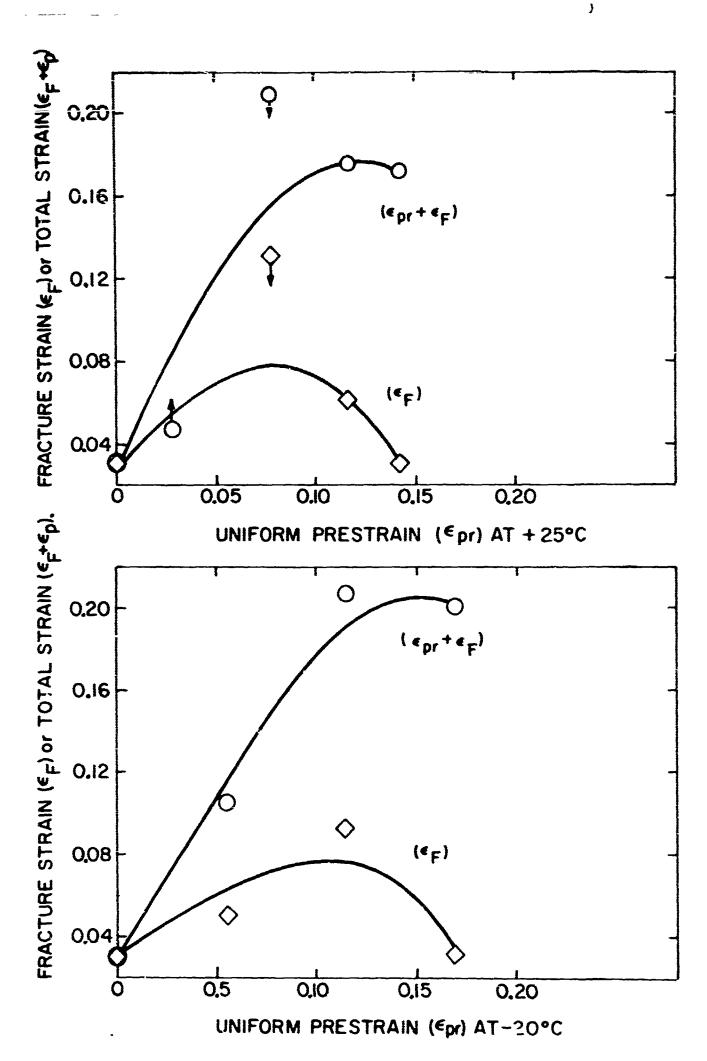


Fig. 14 - Effect of Prestraining Mo-E3 at -20°C and +25°C on Fracture Strain ( $\epsilon_F$ ) and Total Strain ( $\epsilon_{pr} \pm \epsilon_F$ ) at -106°C.

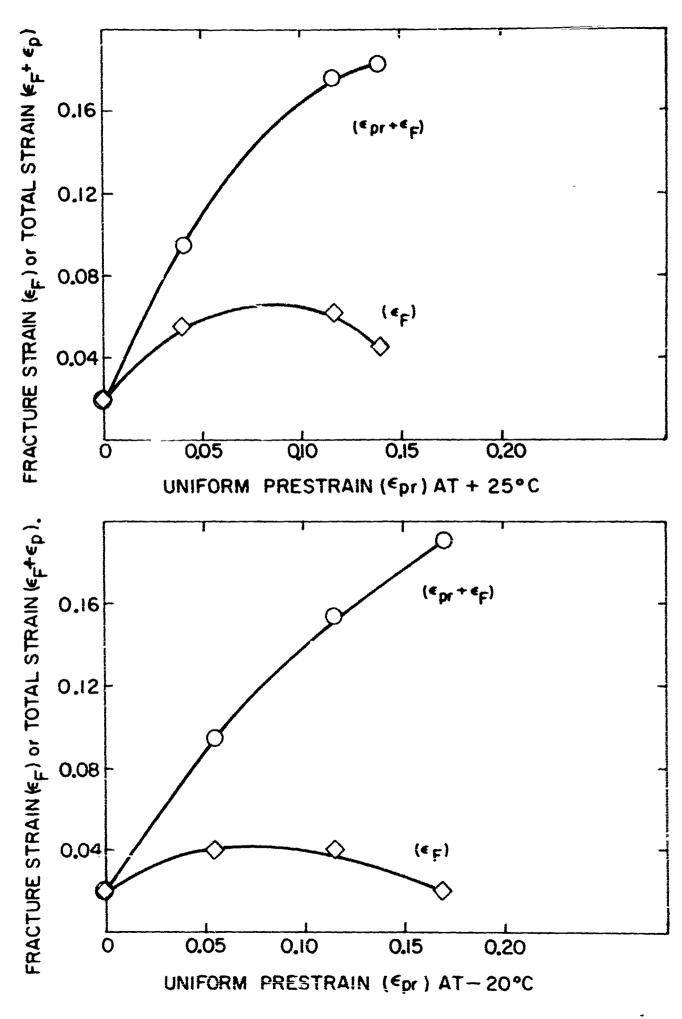


Fig. 15 - Effect of Prestraining Mo-E3 at -20 $^{\circ}$ C and +25 $^{\circ}$ C on Fracture Strain ( $\epsilon_{\rm F}$ ) and Total Strain ( $\epsilon_{\rm pr}$  +  $\epsilon_{\rm F}$ ) at -150 $^{\circ}$ C.

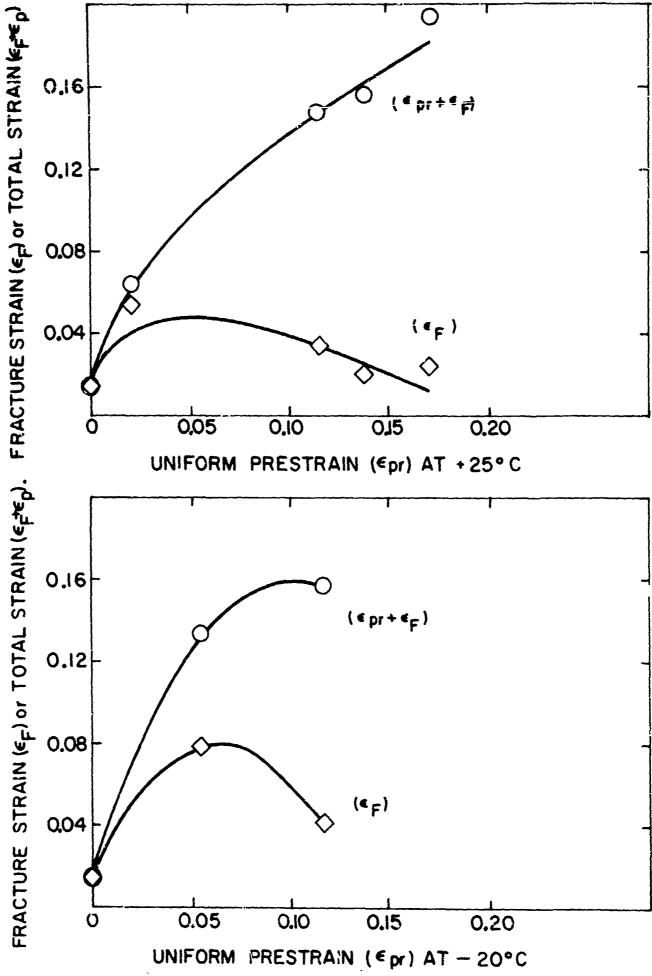


Fig. 16 - Effect of Prestraining Mo-E3 at  $-20^{\circ}$ C and  $+25^{\circ}$ C on Fracture Strain ( $\epsilon_F$ ) and Total Strain ( $\epsilon_F + \epsilon_F$ ) at  $-196^{\circ}$ C.

Table 6

Variation of Fracture Stress of Mo-E3 With Total Strain

Test Tem Prestrain OC	perature Fracture OC	Prestrain (\varepsilon_p)	Total Strain  (  F  pr)	Fracture Stress $(\sigma_F)$	Ratio* of Fracture Stresses With and Without Prestrain
-20°C	-100°C	0.015 0.05 0.085 0.13	0.03 0.05 0.10 0.15 0.20	138 141 151 158 164	1.06 1.08 1.13 1.15
-20°C	-150°C	0.015 0.055 0.11 0.185	0.05 0.10 0.15 0.20	177 180 178 161	1.35 1.34 1.29 1.14
-20°C	-196°C	0.01 0.05 0.115 0.21	0.05 0.10 0.15 0.20	202 193 166 160	1.54 1.44 1.20 1.11
-	-80°C	~	0.03	130	-
-	-79°C	-	0.05	131	-
-	-86°C	-	0.10	134	-
-	-73°C	-	0.15	138	-
-	-70°C	-	0.20	141	-
-	-67°C	-	0.25	144	-

<sup>\*</sup> Values of fracture stress without any prestrain are given in this table for the temperature range of -80 to -67°C.

Table 7

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Effect of Necking Prestrains and Simulated

Necks on the Fracture Stress of Mo-E3 at -100°C

Total Strain	0.03	0.47	0.74	0		0	
Fracture Strain at -100°C	0.03	0, 18	0.10	0		0	
Fracture Stress at -100°C ksi	140	170	173	98		123	
Procedure Prior to test at -100°C	no prestrain	necking prestrain	necking prestrain	prestrained 0.64 and	recrystallized 0.5 hr. at 1200°C	machined with neck (in rolling	praise section) equivalent to 0.64 strair and recrystallized 0.5 hr at 1200°C.
Amt. of Prestrain at +25°C	0	0.29	. 44			* * 0	

Strip was in worked condition prior to No prestrain given after recrystallization, No prestrain given after recrystallization, machining and recrystallizing.

\* \*

-السر ال

Table 8
Fracture Toughness of Mo-E3 at -100°C

	Hartbow	er (W/A)	<u>Irwin G</u> c			
Condition	in-lbs/in <sup>2</sup>	ergs/cm <sup>2</sup>	in-lbs/in <sup>2</sup>	ergs/cm <sup>2</sup>		
as-recrystallized	110	1.9x10 <sup>7</sup>	120	2. 1×10 <sup>7</sup>		
pulled 6% in tension at +25°C	55	9.6x10 <sup>6</sup>	120	2.1x10 <sup>7</sup>		
reduced 6% by rolling	25	$4.4 \times 10^6$	85	1.5×10 <sup>7</sup>		

Assuming that the geometrical effect of necking is to cause a decrease of 30%, it appears that the necking prestrain per se effectively increases  $\sigma_F$  by about 60%.

# 4. Slow Bend Tests at -100°C

Slow bend tests were carried out on Mo-E3 in the following conditions: a) as-recrystallized, b) deformed 6% at +25°C by tensile pulling, c) deformed 6% at +25°C by rolling. V-notch Charpy specimens were machined to the following dimensions: 0.030 inch thick, 0.394 inch wide, and 2 1/8 inches long.

A 0.079 inch deep, 45° V-notch was machined perpendicular to the rolling direction of the strip (parallel to length direction of specimen). These specimens were precracked by room temperature cyclic bending in compression from zero to about 50 ksi (calculated maximum compressive fiber stress at root of the V-notch). The average depth of a fatigue crack as obtained by this procedure was about 0.025 inch. Slow bend tests were carried out at -100°C and measurements of load vs deflection were obtained.

Both the Hartbower and Irwin methods were used to determine resistance to crack propagation from the slow bend test measurements. The Hartbower (9) method of determining the fracture toughness parameter W/A involves dividing the energy (W) corresponding to the integral of the load vs deflection curve by the area (A) of the fractured surface (below the fatigue crack). The Irwin (10) method of determining the fracture toughness parameter  $G_c$  involves use of the following relation:

$$G_{c} = \frac{\left(P_{m}\right)^{2} \frac{d(M^{-1})}{da}}{(19)}$$

where P<sub>m</sub> = maximum load

B = specimen thickness

M = spring constant (P/e) where e = deflection

a = notch depth

An approximate value of dM<sup>-1</sup>/da was obtained from the initial slopes of the P vs e plots corresponding to different initial crack lengths (a).

The fracture toughness values determined from the slow bend tests at  $-100^{\circ}$ C are given in Table 8. The W/A and G<sub>c</sub> values for the as-recrystallized condition show good agreement. However, although decreases in (W/A) of 50 to 75% resulted from plastic strains of 0.06 at  $+25^{\circ}$ C, the corresponding decreases in G<sub>c</sub> are only 0 to 30%. The fracture toughness values at  $-100^{\circ}$ C fall in the range of 25 to 120 in-lbs/in<sup>2</sup> or 0.4 to 2.1 x  $10^{7}$  ergs/cm<sup>2</sup>.

### C. Flow and Fracture Characteristics of Mo-E4 Strip

The effect of test temperature on the tensile properties of Mo-E4 strip is shown in Fig. 17. For this high oxygen material of fine grain size (f = 0.020 mm),  $T_b$  and  $T_d$  appear to coincide at about -35°C, and  $T_m$  occurs at about +25°C. These are higher temperatures than found for the fine grain Mo-E2 and Mo-E3 materials (Table 3). The Mo-E4 also differs from the Mo-E2 and Mo-E3 materials in that  $\epsilon_F \approx 0$  below  $T_d$  and  $T_d$  reaches a constant value at about -100°C. No evidence was found that twinning is responsible for the levelling-off in  $T_d$ . Therefore, this phenomenum remains unexplained.

As shown in Table 9, prestraining the Mo-E4 at +25°C was found to result in increases in fracture stress of about 14 and 20% at -100 and -196°C respectively. The specimens were observed to fracture without any additional plastic strain at these test temperatures. This differs from the Mo-E3 behavior after prestraining since an appreciable amount of strain occurred prior to fracture at -100°C.

The variations of the strain hardening exponent (n) and strain hardening coefficient (K) of Mo-E4 strip with test temperature in the range of -25 to +50°C are shown in Fig. 18. The strain hardening exponent (n) of Mo-E4 decreases markedly with decrease in test temperature, which is opposite to that found for the Mo-E2 and Mo-E3 materials. On the other hand, the strain hardening coefficient (K) increases with decrease in test temperature similar to the Mo-E2 and Mo-E3 materials.

Using the  $\epsilon_F$ , n, and K values for Mo-E4 shown in Figs. 17 and 18 respectively, calculations were made of the uniform fracture stress ( $\sigma_{fs}$ ) and the substructural strengthening factor ( $\sigma_{fs}$ ). As shown in Table 4,  $\sigma_{fs}$  for Mo-E4 was found to be considerably higher than for the Mo-E2 and Mo-E3 materials in the range of 0°C to 25°C. This is attributed to the relatively high strain hardening characteristics of Mo-E4, presumably due to its high oxygen content. The predicted values of  $\sigma_{fs}$  for Mo-E4 under actual necking conditions are shown in Table 5. The predicted values were found to be 27 to 37% higher than the observed values of  $\sigma_{fs}$  in the range of -25 to +50°C. The agreement is relatively poor as compared to that obtained for the Mo-E2 and Mo-E3 materials. This may be due to a difference between high and relatively low oxygen materials with respect to the degree of plastic constraint for the same reduction in area.

### D. Fractographic Characteristics

### 1. Broken Mo-E2 Tensile Specimens

Based on light and electron microscopic observations, the fractographic characteristics of the fine, medium, and coarse grain Mo-E2 tensile specimens were determined. As shown in Figs. 19 to 22, the fractures of the fine grain Mo-E2 specimens tested at +25 to -30°C consist of both cleavage (transgranular) facets and intergranular facets. The cleavage facets contain what are called cleavage steps or river markings, which indicate the direction of crack propagation. On the other hand the intergranular facets

Table 9

1

Effect of Prestrain on the Fracture Stress of Mo-E4 Strip at-100°C and -196°C

Ratio of Fracture Stresses With and Without Prestrain	1.14	1.20
Fracture Stress (after prestrain)	140	148
Fracture Strain (after prestrain)	0	0
Test Temp.	-100	961-
Amount of Prestrain at +25°C	0.28	

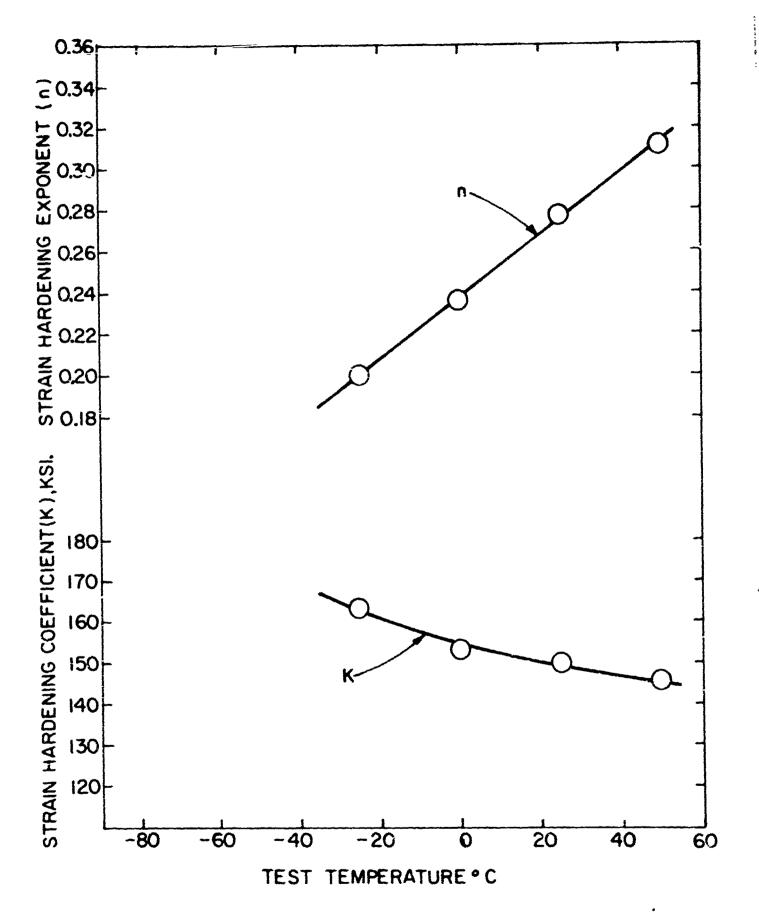


Fig. 18 - Variation of Strain Hardening Exponent (n) and Strain Hardening Coefficient (K) with Test Temperature for Recrystallized Molybdenum (Mo-E4) Strip (f = 0.020 mm).

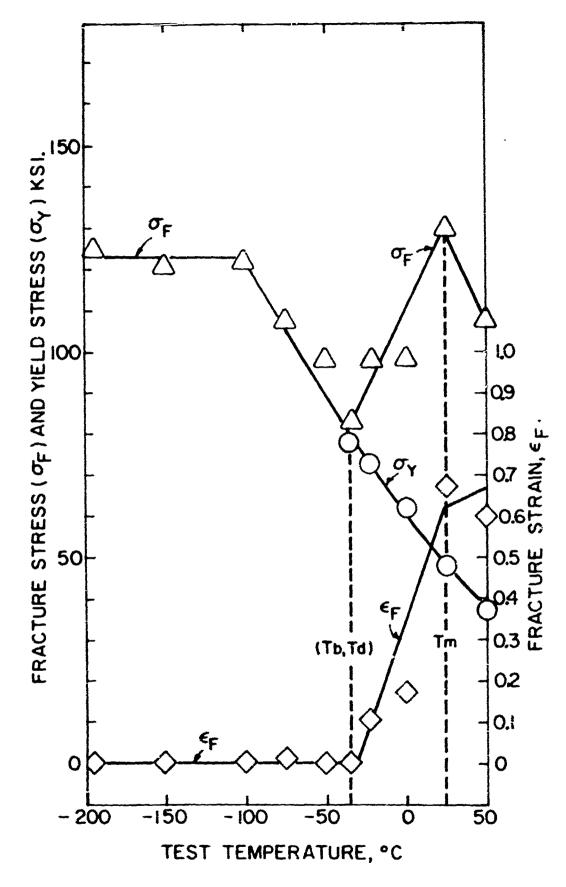


Fig. 17 - Effect of Test Temperature on Tensile Properties of Recrystallized Molybdenum (Mo-E4) Strip (! = 0.020 mm).



Light Fractograph

750X

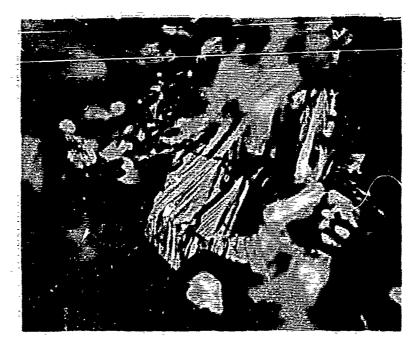
Fig. 19 - Tensile Fracture of Mo-E2 (0.026 mm grain size) at +25°C Showing Several Cleavage Facets (with Cleavage steps) and Two Intergranular Facets (smooth).



Electron Fractograph

2000X

Fig. 20 - Same Specimen as Fig. 19 - Showing Intergranular Facets Which Apparently Contains Fine Precipitates.



Light Fractograph

1000X

Fig. 21 - Tensile Fracture of Mo-E2 (0.026 mm grain size) at -30°C Showing Several Cleavage Facets.



Electron Fractograph

2000X

Same Specimen as Fig. 21 - Showing Cleavage Steps and Smooth Intergranular Facets Which Apparently Contains Fine Precipitates.

are generally smooth, and sometimes contain what appear to be fine precipitates (Figs. 20 and 22). The fractures of the medium grain Mo-E2 was found to be similar to the fine grain Mo-E2. Examples of the coarse grain Mo-E2 fractures in the range of +25 to -196°C are shown in Figs. 23 to 28. At +25°C, the fracture consists predominantly of high distorted cleavage facets (Fig. 23) although a few intergranular facets were observed (Fig. 24). Evidence of what is considered to be initiation of fracture at an intergranular facet at -196°C is shown in Fig. 28.

Table 10 gives a summary of the fractographic characteristics of the Mo-E2 tensile specimens broken in the range of  $\div 25^\circ$  to 196°C. With decrease in test temperature, the relative amount of intergranular facets increases from <1 to about 2% for the fine grain size, from <1 to about 6% for the medium grain size, and from <1 to about 4% for the coarse grain size. The ratio of the average cleavage facet size to the grain size (corrected for total reduction in area prior to fracture) varies from 0.7 to 1.4 for the fine grain size, 0.6 to 1.1 for the medium grain size, and 0.4 to 0.5 for the coarse grain size. The corresponding ratio for the average intergranular facet size varies from 0.7 to 0.9 for both the fine and medium grain size, and 0.1 to 0.3 for the coarse grain size. Thus it appears that although the cleavage and intergranular facet sizes are approximately equal to the grain size for the fine and medium grain size Mo-E2, the cleavage and intergranular facet sizes are significantly smaller than the grain size for the coarse grain Mo-E2.

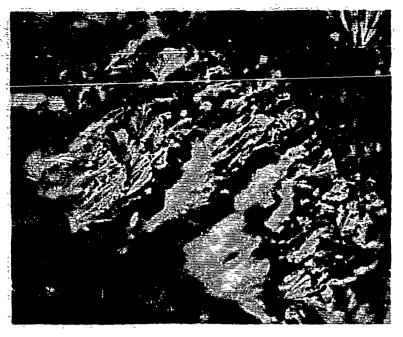
For the coarse grain size Mo-E2, the origin of fracture was traced to a particular intergranular facet from -196°C to T<sub>d</sub>; whereas for the fine and medium grain size Mo-E2, it was traced to a group of both intergranular and cleavage facets. However, above T<sub>d</sub> the origin of fracture of the three grain sizes was traced to a group consisting of only cleavage facets. It is therefore concluded that the mode of fracture initiation in Mo-E2 is intergranular a. T<sub>d</sub> and below, and cleavage above T<sub>d</sub>.

The geometrical location of fracture initiation with respect to the specimen cross section was also determined. It was found that fracture generally initiated in the interior of the specimen. The approximate locations can be expressed by the following coordinates: in the width (w) direction, from w/8 to w/2; and in the thickness (t) direction, from t/4 to t/2. There were only three cases in which fracture initiated at the surface, one at w/3 and the other two at t/2 and t/3.

### 2. Broken Mo-E3 Tensile Specimens

As shown in Figs. 29 and 30, the fractures of the broken fine grain Mo-E3 tensile specimens at -20 and -100°C consist of both cleavage and intergranular facets. Prestraining at +25°C followed by breaking at -100°C results in a greater amount of highly distorted cleavage facets (Figs. 31, 33 and 34) as compared to prestraining at -20°C and breaking at -100°C (Fig. 32). Predominantly intergranular fractures (Figs. 35 and 36) were produced by the simulated necking experiments carried out at -100°C.

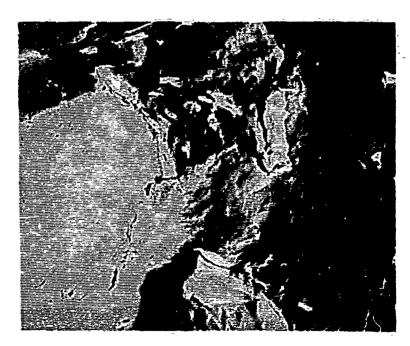
The fractographic characteristics of the Mo-E3 tensile



Light Fractograph

500X

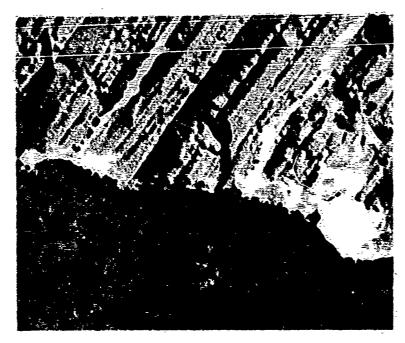
Fig. 23 = Ténsilé Fracture of Mo-E2 (0.174 mm grain size) at +25°C Showing Highly Distorted Cleavage Facets.



Electron Fractograph

2000X

Fig. 24 - Tensile Fracture of Mo-E2 (0.174 mm grain size) at +25°C Showing a Portion of a Smooth Intergranular Facet (Left) Which Apparently Contains Fine Precipitates.



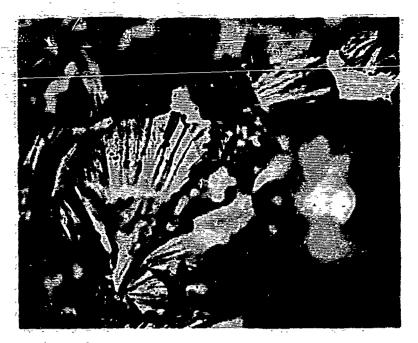
Electron Fractograph

Fig. 25 - Tensile Fracture of Mo-E2 (0.174 mm grain size) at 0°C Showing a Cleavage Facet (upper) and an Intergranular Facet (lower).



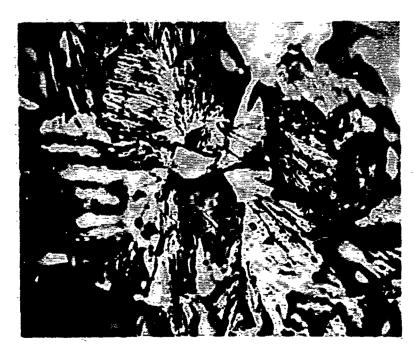
Light Fractograph

Fig. 26 - Tensile Fracture of Mo-E2 (0.174 mm grain size) at -40°C Showing a Cleavage Facet Containing Many Cleavage Steps.



Light Fractograph

Fig. 27 - Tensile Fracture of Mo-E2 (0.174 mm grain size) at -100°C Showing One Intergranular Facet (Smooth) and Several Cleavage Facets.



Light Fractograph

Fig. 28 - Tensile Fracture of Mo-E2 (0.174 mm grain size) at -196°C Showing One Intergranular Facet (Smooth) Surrounded by Several Cleavage Facets with "River" Markings Radiating from Intergranular Facet (Possible Fracture Origin).

Table 10

Fractographic Characteristics of Mo-E2 Broken Tensile Specimens

ion on**	•	t/4)	t/3)	t/2)	<u>3</u>		<u></u>	t/2)	t/4)	t/2)		t/3)		, t/2)	t/3)	(2	t/3)	•	t/4)
Initiation Location**	i	, 2/	`	. 8/	, t	1	,/3	73	7.7	,/3 ,	1	(w/3, t/3)		(w/3 ,	(w/8 ,	t/2	4,		(w/5 ,
re ri	•	5	<u>*</u>	٠.	9:		<u>}</u>	<u>`</u>	` <u>`</u>	· (¥		5		5			<u>`</u> *	•	
Fracture Initiation Mode Location*		cleavage	cleavage	intergran	intergran	ŧ	cleavage	intergran	intergran.	intergran.	ŧ	cleavage	1	cleavage	intergran	intergran.	intergran.	intergran.	intergran,
Average Size mm	0.017	0.019	0.022	0.019	0.022	0.026	0.024	0,035	0.032	0.038	ŧ	0.025	0.035	0.058	0.034	0.043	0.029	0,020	0.025
Intergranular Relative Amount	<b>7</b>	₹		က	7	~	2	~	6	ស	⊽	7	2	2	æ	ĸ	က	ന	₩
e																			
Average Size mm	0.016	0.015	0.033	0.026	0.035	0.018	0.035	0.046	0.033	0.034	0.049	0.042	v. 067	0.078	0.000	0.085	0.085	0.089	0.085
Cleavage Relative Amount	66<	66∧	66	26	86	66<	98	93	91	95	66<	86	86	96	26	26	26	4	96
Fracture Strain, e F	0.98			0.03		1.00	0.55	90.0	0.03	0.01	1.00	0.97	0.46	0.20	0.10	0.05			
Test Temp.	+25	-30	-70	-100	-196	+25	-25	-75	-150	-196	+25	0	-25	-40	-80	001-	-125	-150	-196
Graiņ Size mm	0.018	0.021	0.024	0.026	0.026	0.030	0.035	0.042	0.044	0.044	0.018	0.020	0.139	0.160		0.168	0.170		

\* Corrected for reduction in area prior to fracture.

Location of fracture initiation is given in terms of w and t coordinates which are the width and thickness specimen dimensions respectively. \*\*



Light Fractograph

Fig. 29 - Tensile Fracture of Mo-E3 (0.023 mm grain size) at -20°C Showing Several Cleavage Facets and an Intergranular Facet (Smooth).



Light Fractograph

Fig. 30 - Tensile Fracture of Mo-E3 (0.023 mm grain size) at -100°C Showing Several Cleavage and Intergranular Facets.



Light Fractograph

Fig. 31 - Tensile Fracture of Mo-E3 (0.023 mm grain size)

Uniformly Prestrained (ε = 0.115) at +25°C and

Broken at -100°C Showing Several Highly Distorted

Cleavage Facets.



Light Fractograph

Fig. 32 - Tensile Fracture of Mo-E3 (0.023 mm grain size)
Uniformly Prestrained (€ = 0.110) at -20°C and
Broken at -100°C Showing Several Cleavage Facets.



Light Fractograph

Fig. 33 - Tensile Fracture of Mo-E3 (0.023 mm grain size)

Prestrained by Necking ( $\epsilon = 0.28$ ) at +25°C and

Broken at -100°C Showing Several Cleavage Facets.

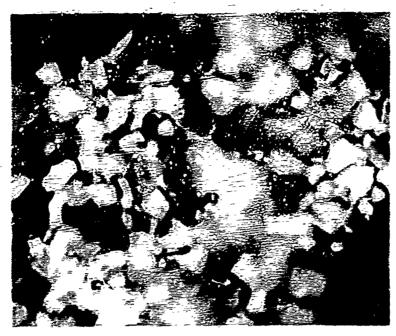


Light Fractograph

Fig. 34 - Tensile Fracture of Mo-E3 (0.023 mm grain size)

Prestrained by Necking ( $\epsilon = 0.64$ ) at +25°C and

Broken at -100°C Showing One Intergranular Facet
(lower right) and Several Cleavage Facets.



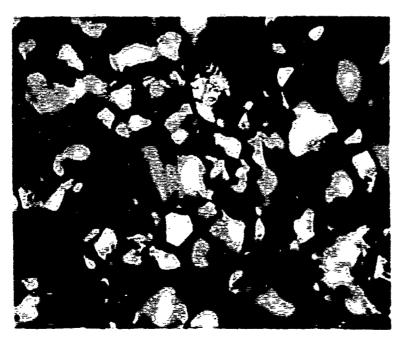
Light Fractograph

Fig. 35 - Tensile Fracture of Mo-E3 (0.023 mm grain size)

Prestrained by Necking ( $\epsilon = 0.64$ ) at  $\pm 25^{\circ}$ C,

Recrystallized, and Broken at  $\pm 100^{\circ}$ C Showing

Predominantly Intergranular Facets.



Light Fractograph

Fig. 36 - Tensile Fracture of Mo-E3 (0.023 mm grain size)
Having Neck Machined Corresponding to a Necking
Prestrain of  $\epsilon = 0.64$ , Recrystallized and Broken
at -100°C Showing Predominantly Intergranular
Facets.

specimens in the range of +25 to -196° C are summarized in Table 11. For specimens that were not prestrained, the fractures are similar to the fine grain Mo-E2 material except that considerably more intergranular facets were found in the Mo-E3 specimens broken at -85 and at -196° C (10 and 40% respectively).

After subjecting the Mo-E3 to a uniform prestrain at either -20 or +25° C and breaking at -100° C or -196° C, the fracture consists of only 5-10% intergranular facets. This represents a considerable decrease in intergranular facets as compared to the non-prestrain results. This would seem to correlate with the higher fracture stress at -100° C as compared to that obtained without prestraining. However, a lower fracture stress was found at -196° C after prestraining.

After a necking prestrain at +25° C followed by breaking at -100° C, the amount of intergranular facets (10-20%) is also lower than that obtained without prestraining. However, simulated necking (as accomplished by forming a neck either by prestraining or machining followed by recrystallization) results in a large increase in the amount of intergranular facets (80-90%) after breaking at -100° C. This correlates with the lower fracture stress at -100° C due to simulated necking as compared to the results for either no prestrain or a necking prestrain.

Similar to the Mo-E2 fine grain size, both the intergranular and cleavage facet size in the Mo-E3 broken tensile specimens are approximately equal to the grain size as corrected for the total reduction in area prior to fracture. This holds for both the non-prestrained and prestrained specimens.

### 3. Broken Mo-E3 Precracked Charpy Slow Bend Specimens

Examples of fractures of Mo-E3 precracked Charpy slow bend specimens broken at -100°C are shown in Figs. 37 and 38, and the observed fractographic characteristics are summarized in Table 12. For tests carried out without prestrain, a greater amount of intergranular facets was found in the bend than in the tensile fracture at -100°C (50 vs 30%). After prestraining about 6% at +25°C either by tensile pulling or by rolling, the bend fracture at -100°C consists of considerably less intergranular facets than without prestraining (5 vs 50%), which is similar to the tensile results (Table 11).

### 4. Broken Mo-E4 Tensile Specimens

Examples of the fractures of broken Mo-E4 tensile specimens are shown in Figs. 39 and 40 and the fractographic characteristics are summarized in Table 13. A sharp change occurs from a predominantly cleavage fracture (<1% intergranular facets) at  $\pm 25^{\circ}$  C to a predominantly intergranular fracture (80% intergranular facets) at  $0^{\circ}$  C. A further increase in the amount of intergranular facets to >99% occurs at  $T_{\rm d}$  (-35°C), and the intergranular fracture remains the same down to -196°C. It was found that fracture at

Table 11

Fractographic Characteristics of Mo-E3 Broken Tensile Specimens

Fracture Initiation	Location**	$(v_1/2, t/4)$	(w/3, 0)	(w/3, t/4)	(w/4, t/3)	(w/8, t/2)	َ د د	-	, t	•	(w/8, t/3)	1	<b>.</b>	(w/2, t/3)	(w/4.0)
Fractur	Mode	cleavage	cleavage	intergran	intergran.	cleavage	cleavage	ŧ	cleavage	cleavage	cleavage	intergran.	intergran.	cleavage	cleavage
LF'acets Average	Size	0.015	0.015	0.012	0.017	0.021	0.020	0.017	0.024	0.013	0.013	0.020	0.019	0.021	0.018
Intergran Facets Relative Averag	Amount %	-	<b>~</b>	10	40	10	ഹ	10	Ŋ	10	Ŋ	06	80	Ŋ	'n
Average	Size	0.016	0.015	0.021	0.021	0.022	0.019	0.025	0.025	0.018	0.016	0.022	0.019	0.026	0.022
Cleavage	Amount 7/0	66	66	06	09	90	95	90	95	90	95	10	20	95	95
Strain(e)	Fracture	1.06	0.91	0.03	0.02	0.08	90.0	0.05	0.08	0		0	0	0.04	0.04
Disatic Strain(6)	Prestrain pr	none	none	none	none	0.077***	0.115***	0.055	0.115***	0.280****	0.640***	****	***	0.113	0.115
\$ 2 4 8 8	Fracture	42.5	-20	יו על	-196	-100	- 100	-100	- 100	-100	- 100	- 100	-100	-196	-196
E E	Prestrain Fractur	1	,	ı	,	+ ሊ	+25	- 20	-20	+25	+25	; ; ·	,	+25	-20
	Grain Size*	2		0.023	0.03	0.020	20.0	0.00	0.022	610.0		0.03	0.00	0.00	0.021

Corrected for reduction in area prior to fracture.

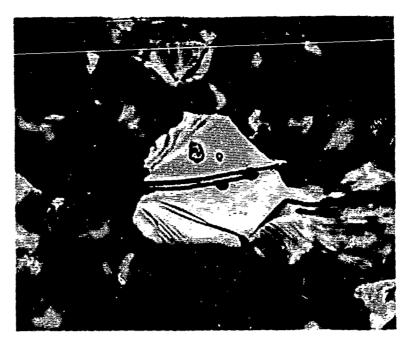
Location of fracture initiation is given in terms of w and t coordinates, which are the width and thickness specimen dimensions respectively. \*

Uniform prestrain. \* \* \*

Necking prestrain. \*\*\*

\*\*\*\*\* Simulated necking by prestraining followed by recrystallization.

\*\*\*\*\* Simulated necking by machining followed by recrystallization.



Light Fractograph

Fig. 37 - Precracked Slow Bend Charpy Fracture of Mo-E3 (0.023 mm grain size) at -100°C Showing a Cleavage Facet Containing a Few Cleavage Steps.



Light Fractograph

Fig. 38 - Precracked Slow Bend Charpy Fracture of Mo-E3 (0.023 mm grain size) Prestrained (6% by rolling) at +25°C and Broken at -100°C Showing Cleavage Facets Containing Many Cleavage Steps.

Fractographic Characteristics of Marrie Broken Precracked Charpy Slow Bend Test Specimens

Fracture Initiation Mode Location	at precrack cleavage at precrack cleavage at precrack
Intergran, Facets Relative Average Amount Size	50 0.017 5 0.013 5 0.015
Cleavage Facets Relative Average Amount Size	50 0.021 95 0.018 95 0.013
Amount of Prestrain Prestrain	none 0.06** 0.06***
Tust Temperature Prestrain Fracture	-100 -100 -100
Tust Prestra	+ + 25 + 25
Grain Size* mm	0.023 0.022 0.022

\* Corrected for reduction in area prior to fracture.

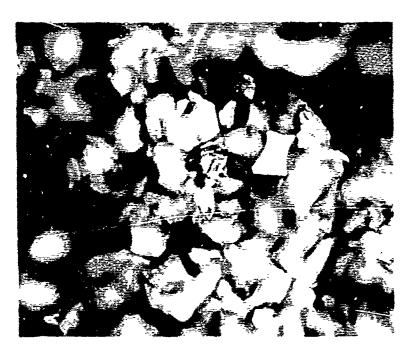
\*\* By tensile pulling.

\*\*\* By rolling.



Light Fractograph

Fig. 39 - Tensile Fracture of Mo-E4 (0.020 mm grain size) at +25°C Showing Several Cleavage Facets.



Light Fractograph

Fig. 40 - Tensile Fracture of Mo-E4 (0.020 mm grain size) at -35°C Showing Two Cleavage Facets and Many Intergranular Facets.

Table 13

# Fractographic Characteristic of Mo-E4 Broken Tensile Specimens

Fracture Initiation Mode Location**	(w/8 , t/3)
	cleavage intergran, intergran. intergran. intergran.
Intergran, Facets Relative Average Amount Size	000000
Cleavage Facets Relative Average Amount Size	0.021 0.019 0.020 0.020 0.016 0.018
Cleavage Relative A Amount	> 99 20 20 1
Plastic Strain	0.67 0.35 0.01 0
<u>ت</u>	0.28*** 0.25***
Test Temperature Prestrain Fracture	+25 0 -35 -100 -196 -100
Test Terr Prestrain	+ + + + + + + + + + + + + + + + + + +
Grain Size* mm	0.014 0.017 0.020 0.020 0.020 0.017

Corrected for reduction in area prior to fracture,

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Location of fracture initiation is given in terms of w and t coordinates which are the final width and the final thickness of the specimen dimensions respectively. \*\* \*\*

Subjected to uniform prestrain at +25° C. \*\* \*\* \*\*

+25°C initiates by a cleavage mode and by an intergranular mode at 0°C. No definite evidence was obtained with respect to the location of fracture initiation at -35 to -196°C; however, it is presumed that the fracture initiation mode is intergranular since only a few cleavage facets were found present. Prestraining at +25°C and breaking at -100°C and at -196°C was found to increase the amount of cleavage facets (50 and 25% respectively), which correlates with the increases in fracture stress obtained by prestraining. Similar to the fine grain Mo-E2 and Mo-E3 materials, both the cleavage and intergranular facet sizes are approximately equal to the grain size as corrected for the reduction in area prior to fracture.

### 5. Other Observations

Examinations were carried out for the occurrence of microcracks and twins in the vicinity of the fractures of the Mo-E2, Mo-E3, and Mo-E4 materials. No evidence of isolated microcracks was found, although branching of secondary cracks from the main fractures was observed. Likewise, no definite evidence of twinning could be found.

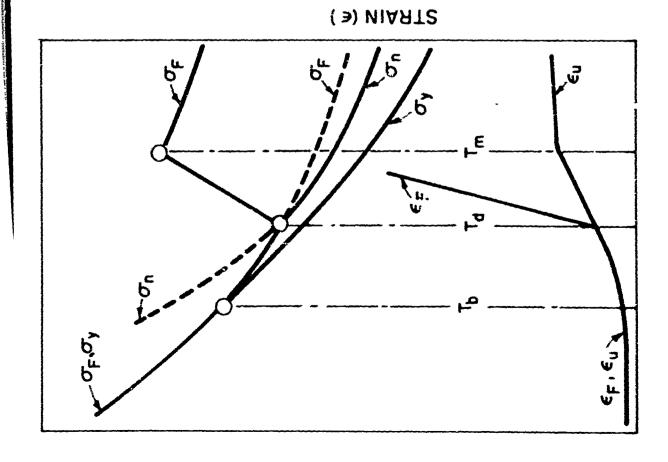
### E. Theoretical Considerations

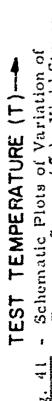
### 1. Relation of Yield Stress to Fracture Stress

As illustrated schematically in Fig. 41, the brittleness transition temperature  $(T_b)$  corresponds to the intersection of curves representing the variation with temperature of the observed fracture stress  $(\sigma_F)$  and a yield stress parameter  $(\sigma_V)$ . For each of the molybdenum strip materials studied, it was found that the values of proportional limit  $(\sigma_{pl})$ , lower yield stress  $(\sigma_{lys})$  or upper yield stress  $(\sigma_{uys})$  corresponding to a given test temperature are within about 15% of each other. Considering that the probable accuracy of measurement to about  $\pm$  10% for these three stress parameters, it does not seem to matter whether  $\sigma_V$  represents  $\sigma_{pl}$ ,  $\sigma_{lys}$  or  $\sigma_{uys}$ . In Figs. 42 to 46, the locations of  $\sigma_{lys}$  for Mo-E2 (3 grain sizes), Mo-E3, and Mo-E4 are shown by the intersections of a band that represents the accuracy of  $\sigma_{lys}$  and  $\sigma_{lys}$ .

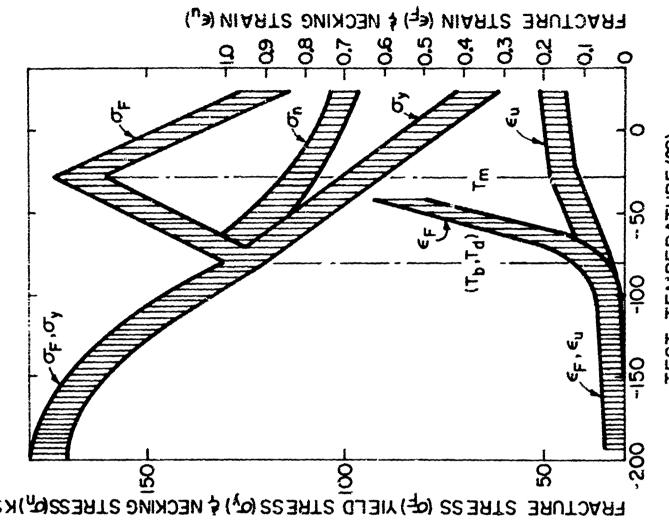
## 2. Relation of Necking Stress at Fracture Stress

Also illustrated schematically in Fig. 41 is the intersection of the observed fracture stress ( $O_F$ ) and the necking stress ( $O_n$ ), where the latter is the true stress corresponding to maximum load. This intersection is shown to occur at the minimum in the  $O_F$  vs. temperature plot. The temperature at which the minimum occurs has been previously defined as the tensile ductility transition temperature ( $T_d$ ). It was hypothesized by Lement (3) that  $O_n$  intersects  $O_F$  at  $T_d$  because below  $T_d$  the elongation at fracture was found to be uniform whereas above  $T_d$  necking occurs prior to fracture. This situation is schematically indicated in Fig. 41 by the intersection of the fracture strain ( $\varepsilon_F$ ) and the maximum uniform strain ( $\varepsilon_U$ ) curves at  $T_d$ .

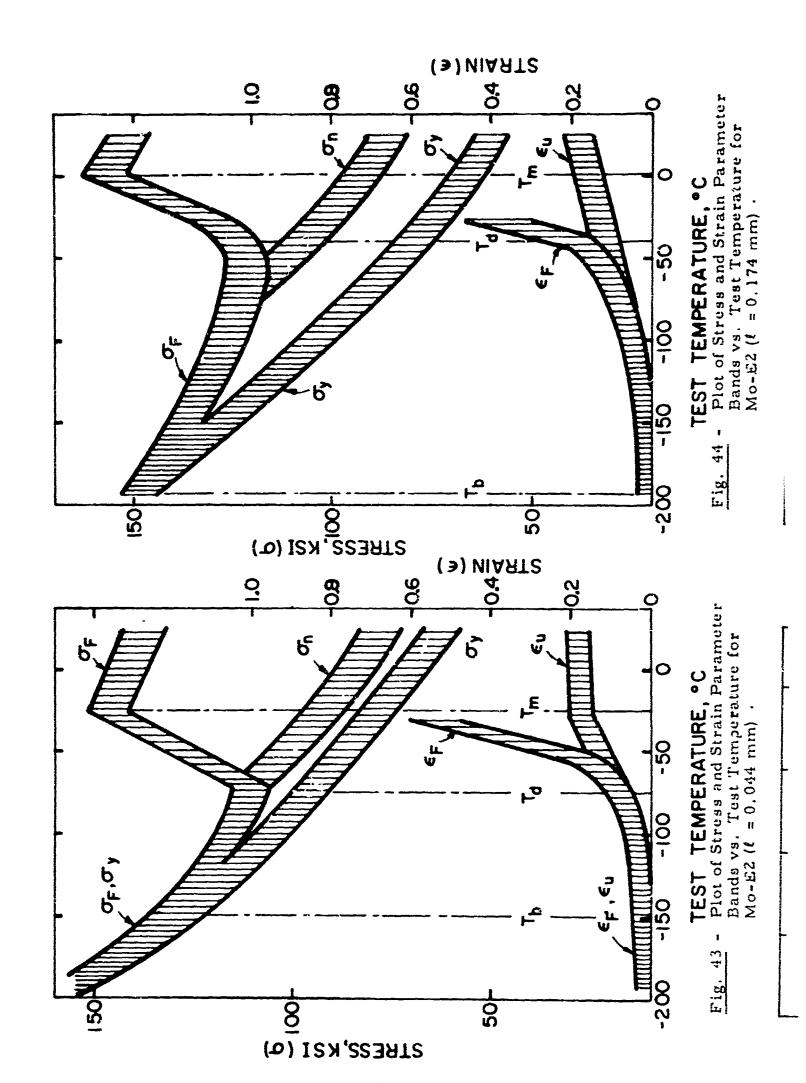


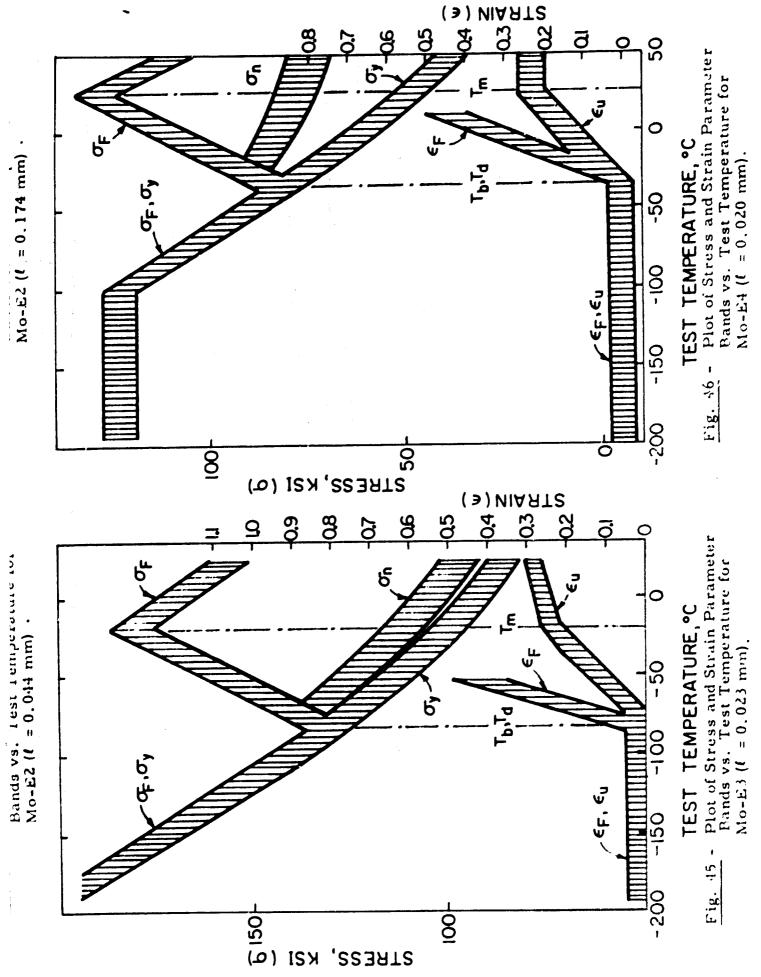


Schematic Plots of Variation of Fracture Stress (\$\sigma\_{\begin{subarray}{c} \empsyserize{\emps



TEST TEMPERATURE (%)
42 - Plot of Stress and Strain Parameter
Bands vs. Temperature for Mo-E2
(f = 0.026 mm).





Bands representing the probably accuracy of  $\sigma_F$ ,  $\sigma_n$ ,  $\varepsilon_F$ , and  $\varepsilon_u$  for the various molybdenum strip materials are shown in Figs. 42 to 46 as a function of test temperature. For the fine grain Mo-E2, No-E3 and Mo-E4 materials,  $T_b$  and  $T_d$  were found to approximately coincide. This is indicated by the intersection of the  $\sigma_y$ ,  $\sigma_n$ , and  $\sigma_r$ , bands at  $\sigma_r$  and  $\sigma_r$  for the intermediate and coarse grain Mo-E2 materials. This is indicated by the fact that the intersection of the  $\sigma_y$  and  $\sigma_r$  bands occurs below that of the  $\sigma_r$  and  $\sigma_r$  bands Figs. 43 and 44. For all the materials studied, the  $\sigma_u$  and  $\sigma_r$  bands intersect at  $\sigma_r$  Figs. 42 to 46.

According to Ault (11), who investigated the tensile properties of a fine grain and relatively impure molybdenum strip material,  $\sigma_{uys}$  intersects  $\sigma_{F}$  at the minimum in the  $\sigma_{F}$  vs. temperature plot (which is defined here as  $\sigma_{d}$ ). Based on the present study, this should occur if  $\sigma_{d}$  and  $\sigma_{d}$  are equal as was found for the fine grain molybdenum strip materials. However, the more general condition for  $\sigma_{d}$  is the intersection of  $\sigma_{d}$  and  $\sigma_{d}$  as indicated by the results for the intermediate and coarse grain strip materials Figs 42 and 43. For these grain sizes,  $\sigma_{d}$  occurs above  $\sigma_{d}$  and  $\sigma_{d}$  intersects  $\sigma_{d}$  at  $\sigma_{d}$ .

### 3. Locking Parameters

### 3.1 Yield Locking Parameter

According to Cottrell (5),  $\sigma_F = \sigma_V$  at  $T_b$  and  $\sigma_F$  is inversely proportional to the yield locking parameter  $(k_y)$  at  $T_b$ . The variation of  $k_y$  with test temperature for Mo-E2 as determined by the Petch grain size method was shown in Fig. 5. An alternative method of determining  $k_y$  is by the Owen (12) extrapolation method. This first involves extrapolating the flow stress curve (beyond the Lüders strain) back to the elastic line to determine the frictional stress ( $\sigma_i$ ). Based on the power law,  $\sigma_i$  at the intersection of the plastic and elastic curves is given by

$$\sigma_{i} = K \left( \frac{\sigma_{i}}{E} \right)^{n} \tag{20}$$

From the Petch relation,

$$k_{y} = \frac{\sigma_{y} - \sigma_{i}}{\sigma_{i} - i/2}$$
 (21)

Using Eq. (20) and (21), calculations were made of  $k_y$  and  $\sigma_i$  at test temperatures of  $+25^{\circ}$  C,  $T_d$ , and  $T_b$  for all of the molydenum strip materials and the results are given in Table 14. The  $T_b$  values of  $k_y$  and  $\sigma_i$  for the intermediate and coarse grain Mo-E2 given in this table are based on rough extrapolations of the K and n curves down to -150 and -196 C

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Table 14

Yield Locking Parameter (ky) and Frictional Stress (Gi) for Mo-E2, Mo-E3, and Mo-E4 Strip

Material and Grain Size	Temp. Temp. G dynes/cm	Strain Hardening Coefficient (K)	Strain Hardening Exponent	By Petch Grain Size Method $ \begin{pmatrix} \sigma_{i} \\ \sigma_{i} \end{pmatrix} \frac{(k_{v})}{dynes/cn} $	By Petch Grain Size Method $ \frac{(\sigma_i)}{(\sigma_i)} \frac{(k_v)}{(k_v)} $ dynes/cm <sup>2</sup> dynes/cm <sup>3/2</sup>	Extrapolation Method $(\sigma_i)$ $(k_y)$ $(k_y)$ dynes/cm <sup>2</sup> dynes/cm	By Owen '  Extrapolation Method (0,) (k,)  dynes/cm <sup>2</sup> dynes/cm <sup>3/z̄</sup>
Mo-E2 (0.026mm)	+25 4.6×10 <sup>9</sup> -80(T <sub>b)</sub> 8.8×10		0.065 0.115	3.9×10 <sup>7</sup> 5.3×10 <sup>7</sup>	4.5×10 <sup>7</sup> 16×10	$4.3\times10^{7}$ $6.8\times10$	1.8×10 <sup>7</sup> 10×10
Mo-E2 (0.044mm)	+25 4.5×10 <sup>9</sup> -75(T <sub>d</sub> ) 7.0×10 <sup>9</sup> -150(T <sub>b</sub> ) 9.3×10 <sup>9</sup>	6.1×10 <sup>9</sup> 12.6×10 <sup>9</sup> 18.2×10 <sup>9</sup>	0.090	3.9×10 <sup>7</sup> 5.2×10 <sup>7</sup> 6.4×10 <sup>7</sup>	4.5×10 <sup>7</sup> 15×10 <sup>7</sup> 24×10 <sup>7</sup>	3.3×10 <sup>7</sup> 6.5×10 <sup>7</sup> 6.7×10 <sup>7</sup>	7.9×10 <sup>7</sup> 3.3×10 <sup>7</sup> 17×10 <sup>7</sup>
Mo-E2 (0. 174mm)	+25 4.3×10 <sup>9</sup> -40(T <sub>d</sub> ) 5.7×10 <sup>9</sup> -196(T <sub>b</sub> ) 10.7×10	6.6×10 <sup>9</sup> 10.0×10 <sup>9</sup> 14.1×10	0.073 0.087 0.22	3.9×10 <sup>7</sup> 4.6×10 <sup>7</sup> 7.1×10	4.5×10 <sup>7</sup> 12×10 <sup>7</sup> 29×10 <sup>7</sup>	3.9×10 <sup>7</sup> 5.4×10 <sup>7</sup> 5.6×10	$4.1\times10^{7}  4.1\times10^{7}  68\times10^{7}$
Mo-E3 (0.023mm)	+25 5.7×10 <sup>9</sup> -80(T <sub>b</sub> ) 9.3×10 <sup>9</sup>	7.1×10 <sup>9</sup>	0.048	r 1	2.2×10 <sup>7*</sup> 7.0×10 <sup>7*</sup>	5.3×10 <sup>7</sup> 8.5×10 <sup>7</sup>	1.1×10 <sup>7</sup> 3.5×10 <sup>7</sup>
Mo-E4 (0.020mm)	+25 3.4×10 <sup>9</sup> -35(T <sub>b</sub> ) 5.9×10 <sup>9</sup>	10.7×10 <sup>9</sup>	0.276		19×10 <sup>7*</sup> 22×10 <sup>7*</sup>	1.3×10 <sup>7</sup> 3.4×10 <sup>7</sup>	9. 4×10 <sup>7</sup>

\* Approximate grain size k value.

respectively. Corresponding values are given in Table 14 for  $k_y$  and  $\sigma_i$  as determined by the Petch grain size method for the Mo-E2 materials (Figs. 4 and 5), which also required extrapolation down to -196° C. For the Mo-E2 materials, relatively good agreement (within 30%) was found between the  $\sigma_i$  values as determined by the Owen extrapolation and the Petch grain size methods. On the other hand, there are much larger discrepancies between the  $\kappa_i$  values as obtained by these two methods. Furthermore, the extrapolation  $\kappa_i$  values exhibit anomalous variations with test temperature.

Since the Petch grain size  $k_y$  values appear to be more reasonable, it was decided to use these values in subsequent calculations of the effective surface energy for crack initiation. However, this raised the difficulty that only the Owen extrapolation  $k_y$  values were determined for the Mo-E3 and Mo-E4 materials. As an approximation, it was decided to use the ratio of the grain size  $k_y$  to the extrapolation  $k_y$  for the fine grain Mo-E2 in order to convert the extrapolation  $k_y$  values for the fine grain Mo-E3 and Mo-E4 materials to grain size  $k_y$  values. For the fine grain Mo-E3, this ratio is about 2; and values of grain size  $k_y$  for Mo-E3 and Mo-E4 as converted on this basis are given in Table 14.

# 3.2 Flow Locking Parameter

The flow locking parameter  $(k_f)$  was determined as a function of plastic strain  $(\epsilon_p)$  and test temperature for Mo-E2. This involved determination of the variation with grain size of the uniform flow stress  $(\mathcal{O}_{fs})$  as determined by the power law:

$$\sigma_{fs} = K \left( \epsilon_{p} \right)^{n}$$
 (22)

In Figs. 47 and 48, the values of  $O_{fs}$  corresponding to  $\epsilon_p$  values of 0.2 to 1.0 are plotted as a function of the grain size parameter ( $\ell^{-1/2}$ ) for test temperatures of -50, -15, and +25° C. The slopes of these  $O_{fs}$  vs  $\ell^{-1/2}$  plots give  $k_f$  as defined by the following relation:

$$k_{f} = \frac{\sigma_{fs} - \sigma_{o}}{e^{-1/2}}$$
 (23)

Where  $\sigma_o$  is a constant for a given value of  $\epsilon_p$  and temperature. As is apparent from Figs 47 and 48, the value of  $k_f$  for a given strain and temperature is not significantly affected by plotting  $\sigma_{fs}$  against the final rather than the initial grain size.

Values of kf based on Fig. 47 are given in Table 15. For each  $\epsilon_p$ ,  $k_f$  is approximately equal to  $k_y$  at  $+25^{\circ}$  C and with decrease in test temperature goes through a minimum at about  $-15^{\circ}$  C. The ratio of  $k_f$  to  $k_y$  was found to decrease from about 1.0 to about 0.4 in the range of +25 to  $-15^{\circ}$  C, and from -15 to  $-50^{\circ}$  C there is little change. These results differ from the work of Armstrong (13), who reported that  $k_f/k_y$  equals about 0.3 for molybdenum at room temperature.

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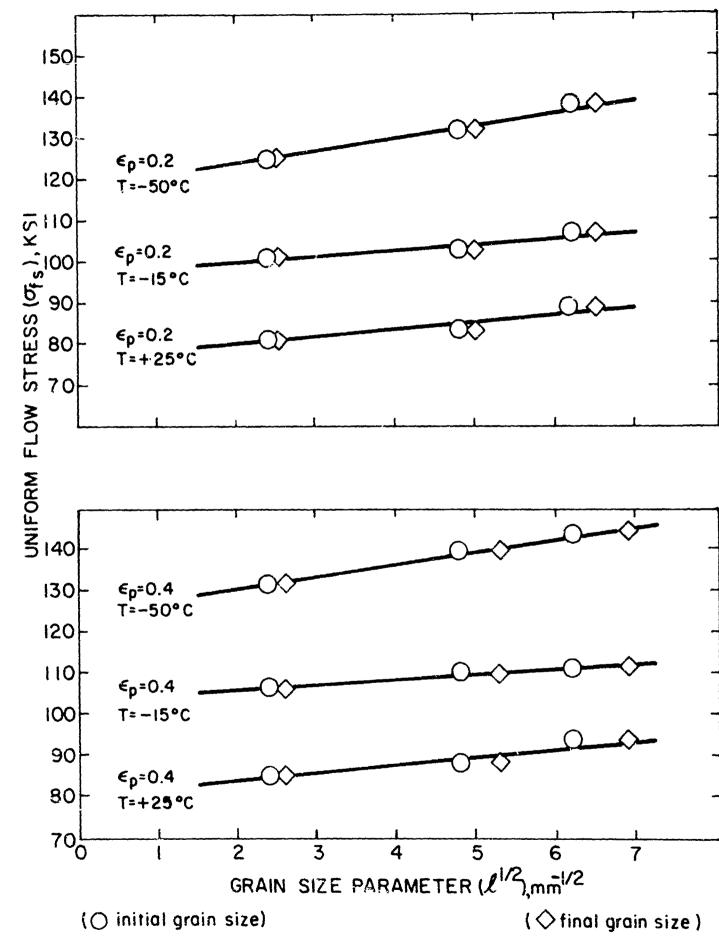


Fig. 47 - Plots of Uniform Flow Stress (0, ) vs. Grain Size Parameter for Recrystallized Mo-E2 Strip at Plastic Strains ( $\epsilon$ ) of 0.2 and 0.4.

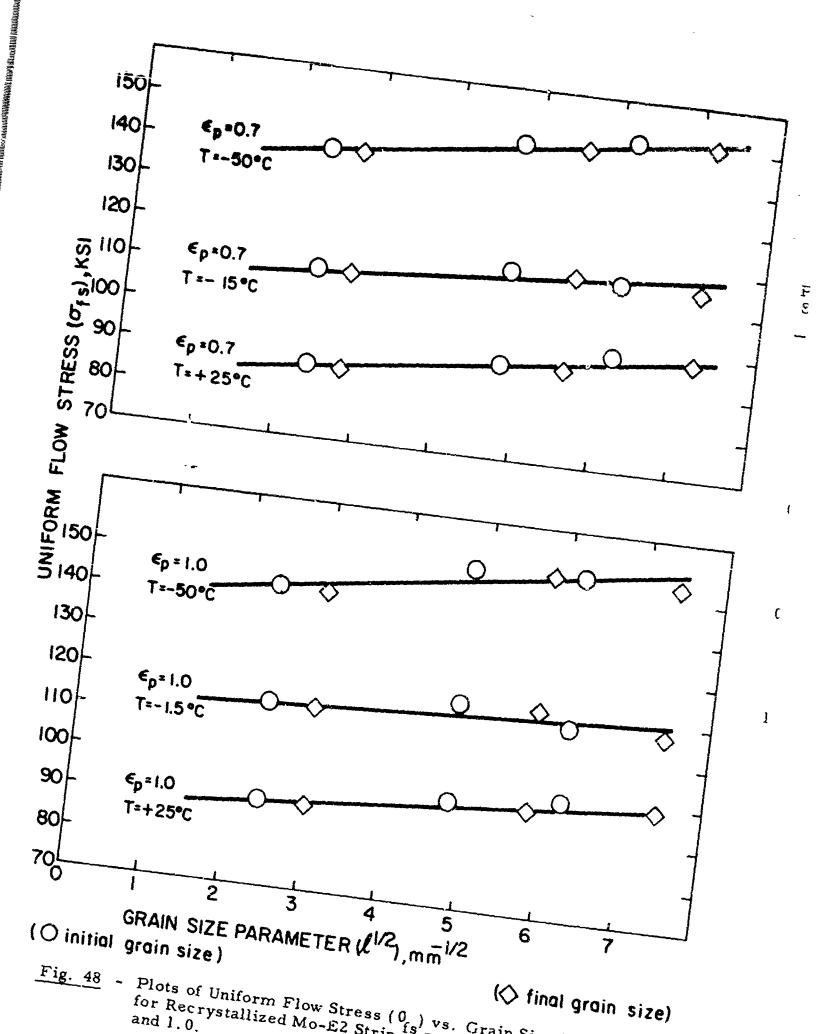


Fig. 48 - Plots of Uniform Flow Stress (0) vs. Grain Size Parameter for Recrystallized Mo-E2 Strip at Plastic Strains ( $\epsilon_p$ ) of 0.7

Table 15
Flow Locking Parameter (kf) for Mo-E2 Strip

Plastic Strain (ep)	Test Temp. (T)	Flow Locking Parameter (k <sub>f</sub> ) dynes/cm <sup>3/2</sup>	Yield Locking Parameter (k <sub>y</sub> ) dynes/cm <sup>3/2</sup>	Ratio k <sub>f</sub> /k y
0.2	+25	6.0×10 <sup>7</sup>	4.5x10 <sup>7</sup>	1.3
	-15	3.5x10 <sup>4</sup>	8.0x10 <sup>7</sup>	0.4
	-50	$4.0 \times 10^{7}$	13×10 <sup>7</sup>	0.3
0.4	+25	4.5x10 <sup>7</sup>	4.5x10 <sup>7</sup>	1.0
	-15	$3.0 \times 10^{7}$	$8.0 \times 10^{7}$	0.4
	-50	6.5x10 <sup>7</sup>	13×10 <sup>7</sup>	0.5
0.7	+25	5.0x10 <sup>7</sup>	4.5x10 <sup>7</sup>	1.1
	-15	$3.0 \times 10^{7}$	$8.0 \times 10^{7}$	0.4
	-50	5.5x10 <sup>7</sup>	13×10 <sup>7</sup>	0.4
1.0	+25	$4.0 \times 10^{7}$	4.5x10 <sup>7</sup>	0.9
1.0	-15	2.5x10 <sup>7</sup>	$8.0 \times 10^{7}$	0.3
		6.6x10 <sup>7</sup>	13×10 <sup>7</sup>	
	-50	o.oxiu	UIXCI	0.5

Values of  $k_f$  were not determined for the Mo-E3 and Mo-E4 materials because these were recrystallized to a fine grain size only. However,  $k_f$  for these materials can be approximated by using the  $k_f/k_y$  ratio determined for Mo-E2 and the  $k_y$  values for Mo-E3 and Mo-E4 given in Table 14 (converted from extrapolation to grain size  $k_y$  values). Approximate values of  $k_f$  for Mo-E3 and Mo-E4 as determined on this basis are given in Table 16. These values also show a minimum at -15° C.

### 4. Calculation of Effective Surface Energy for Crack Initiation

The Cottrell (5) fracture relation for crack initiation at the brittleness-transition temperature  $(T_b)$  is as follows:

$$\gamma_i = \frac{k_y \ell^{1/2} \sigma_F}{8G} \tag{24}$$

where  $\gamma_i^{i}$  = effective surface energy for crack initiation

k = yield locking parameter based on normal stress and full grain diameter

G = shear modulus and is equal to  $\frac{E}{2(1 + \nu)}$  where  $\nu$  is Poisson's ratio (0.3)

full grain diameter

The effective surface energy for crack initiation ( $\gamma_i$ ) in Mo-E2, Mo-E3 and Mo-E4 at the brittleness transition temperature ( $T_b$ ) was calculated using a) values of  $G_F$  at  $T_b$  given in Figs. 1, 2, 3, 11 and 17; b) values of  $k_y$  at  $T_b$  based on the Petch grain size method (Table 14); and c) values of G at  $T_b$  as determined from the variation of the elastic modulus (E) with temperature for molybdenum (Fig. 49), i.e. G = 0.38 E. Calculations were also made of  $\gamma_i$  at temperatures above  $T_b$  based on a modification of Eq. (4) which involves substituting the flow locking parameter ( $k_f$ ) for  $k_y$  in order to take into account the relatively large plastic strains that occur prior to fracture. Values of  $k_f$  were obtained from Tables 15 and 16, and the results of  $\gamma_i$  calculations are given in Table 17.

As shown in Table 17, the values of  $\gamma_i$  at  $T_b$  (based on  $k_y$ ) are in the range of 0.7 x  $10^4$  to 3.5 x  $10^4$  ergs/cm<sup>2</sup>. With increase in temperature, an initial decrease in  $\gamma_i$  (based on  $k_f$ ) occurs which is a mathematical consequence of  $k_f$  being lower than  $k_y$ . Since a lower value of  $\gamma_i$  would be expected if the initiation mode is intergranular as compared to cleavage, this casts doubt on the validity of Eq. (24) based on  $k_y$ . Above  $T_d$ ,  $\gamma_i$  for Mo-E2 and Mo-E3 ramains essentially constant up to at least +25°C and the mode of crack initiation is cleavage over this range. On the other hand, there is about a 75% increase in  $\gamma_i$  for Mo-E4 between 0°C and +25°C where the initiation mode changes from integranular to cleavage. It therefore appears that the high oxygen content of Mo-E4 results in different  $\gamma_i$  as well as fracture initiation characteristics than the relatively low oxygen content materials (Mo-E2 and Mo-E3)

<u>Table 16</u>
Approximate Values of Flow Locking Parameter (kf) for Mo-E3 and Mo-E4 Strip

<u>Material</u>	Plastic Strain (e <sub>p</sub> )	Test Temp. (T) C	Approx. k <sub>f</sub> /k *	Approx.  k **  dynes/cm <sup>3</sup> /2	Approx.  k f dynes/cm <sup>3/2</sup>
Mo-E3	0.2-1.0	+25 -15	1.0 0.4	$2.2 \times 10^{7}$ $3.9 \times 10^{7}$	2.2x10 <sup>7</sup>
		-50	0.4	5.3x10 <sup>7</sup>	2. 1x10 <sup>7</sup>
Mo-E4	0.2-1.0	+25	1.0	19x10 <sup>7</sup>	19×10 <sup>7</sup>
		-15	0.4	21x10 <sup>7</sup>	$8.4 \times 10^{7}$
		-50	0.4	23x10'	9.0x10'

<sup>\*</sup> Assumed to be the same as for Mo-E2 strip given in Table 15.

<sup>\*\*</sup> Approximate grain size k values given in Table 14 and linear extrapolations.

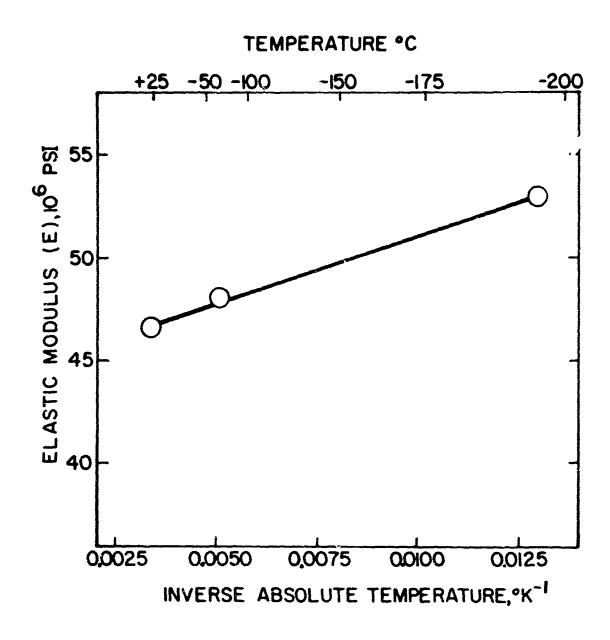


Fig. 49 - Variation of Elastic Modulus of Molybdenum (Mo-El Rod) with Temperature.

Calculated Effective Surface Energy for Crack Initiation in Mo-E2, Mo-E3 and Mo-E4 Strip

•	cleavage cleavage	intergran, intergran	cleavage cleavage cleavage	intergran.	cleavage cleavage cleavage
	0.2×10 <sup>4</sup>	1.4×10 <sup>4</sup> 0.25×10 <sup>4</sup>	0.25×10 <sup>4</sup> 0.25×10 <sup>4</sup> 0.25×10 <sup>4</sup>	3.5 x10 <sup>4</sup> 0.4x10 <sup>4</sup>	0.4×10 <sup>4</sup> 0.4×10 <sup>4</sup> 0.5×10 <sup>4</sup>
Locking Parameter (k <sub>1</sub> ) or(k <sub>f</sub> )  dynes/cm 3/ 16x10 <sup>7**</sup> 6.5x10 <sup>7</sup> 4.3x10 <sup>7</sup>	4.5×10 <sup>7</sup>	$2.4 \times 10^{7 **}$ $4.5 \times 10^{7}$	$4.0 \times 10^{7}$ $3.7 \times 10^{7}$ $4.5 \times 10^{7}$	2 9×10 <sup>7</sup> ** 3.8×10 <sup>7</sup>	3.1×10 <sup>7</sup> 4.5×10 <sup>7</sup>
Fracture Stress ( $\sigma_{\rm F}$ ) dynes/cm $^2$ 8.6×10 $^9$ 10.0×10 $^9$ 11.6×10 $^9$		9.0×10 <sup>9</sup>	8.8×10 <sup>7</sup> 10.1×10 <sup>9</sup> 9.6×10 <sup>9</sup>	$10.3 \times 10^9$ $8.4 \times 10^9$	
Grain Size** mm 0.026	0.018	0.044	0.034 0.034 0.030	0.174 0.160 0.134	0.120
ซ	1.2×10 <sup>1.2</sup>	1.3×10 <sup>12</sup> 1.25×10 <sup>12</sup>	1.2×10 <sup>1.2</sup> 1.2×10 <sup>1.2</sup>	1.4×10 <sup>12</sup> 1.2×10 <sup>12</sup> 1.2×10 <sup>12</sup>	1.2×10 <sup>12</sup> 1.2×10 <sup>12</sup>
Fracture Strain (F) 0.04 0.35	0.98	0.02	0.65	0.02 0.17 0.62	0.95
Test Temp. °C -80 -55	+ + - + -	-150 -75 -50	- 25 + 25	-196 -40 -20	+52 +58
Material and Original Grain Size Mo-E2 (0.026 mm)	73	(0.044 mm)		Mo-E2 (0. 174 mm)	

Table 17 (Continued)

(D) DETERMINE CONTRACTOR OF THE PARTY OF THE	Effective Surface Energy for Crack Initiation in Mo-E2, Mo-E3 and Mo-E4 Strip
	Calculated Effective Sur

Fracture Initiation Mode	intergran, cleavage cleavage cleavage	intergran. intergran. intergran. cleavage
Effective Surface Energy $(\gamma_i)$ ergs/cm <sup>2</sup>	0.3×10 <sup>4</sup> 0.1×10 <sup>4</sup> 0.1×10 <sup>4</sup> 0.1×10 <sup>4</sup>	0.8×10 <sup>4</sup> 0.25×10 <sup>4</sup> 0.4×10 <sup>4</sup> 0.7×10 <sup>4</sup> 0.55×10 <sup>4</sup>
Locking Parameter (k <sub>v</sub> )or(k <sub>f</sub> ) dynes/cm <sup>3/</sup> 2	7.0×10 <sup>7</sup> ** 2.0×10 <sup>7</sup> 1.7×10 <sup>7</sup> 2.2×10 <sup>7</sup>	22×10 <sup>7</sup> ** 8.6×10 <sup>7</sup> 12×10 <sup>7</sup> 19×10 <sup>7</sup>
Fracture Stress $(\sigma_{\overline{F}})$	9.0×10 <sup>9</sup> 11.2×10 <sup>9</sup> 12.4×10 <sup>9</sup> 10.7×10 <sup>9</sup>	5.5x10 <sup>9</sup> 6.2x10 <sup>9</sup> 7.6x10 <sup>9</sup> 9.0x10 <sup>9</sup> 7.5x10 <sup>9</sup>
Grain Size*	0.023 0.018 0.016 0.015	0.020 0.019 0.017 0.015
Shear Modulus (G) dynes/cm <sup>2</sup>	1.25×10 <sup>12</sup> 1.25×10 <sup>12</sup> 1.2×10 <sup>12</sup> 1.2×10 <sup>12</sup>	1. 2×10 <sup>12</sup>
Fracture Strain (F)	0.03 0.54 0.91 1.06	0.00 0.10 0.36 0.68 0.61
Test Temp.	-85 · -45 -20 +25	-35 -25 0 +25 +50
Material and Original Grain Size	Mo-E3 (0.023 mm)	Mo-E4 (0.020 mm)

\* Corrected for reduction in area prior to fracture.
\*\*

Yield locking parameter  $(k_y)$  values; other values given in this column are flow locking parameter  $(k_f)$ 

#### 5. Effective Surface Energy for Crack Propagation

#### 5.1 Based on Tensile Tests

Assuming that fracture of the molybdenum materials is controlled by crack initiation rather than crack propagation, it follows that using the value of the observed fracture stress in a Griffith-Orowan type of relation should give an upper limit to the probable value of the effective surface energy for crack propagation  $(\gamma_p)$ . Accepting this restriction, calculations were made of  $\gamma_p$  under plane strain conditions using the Irwin (14) relation for an interior crack of radius c that is small with respect to the section area:

$$\gamma_{\rm p}' = \frac{4(1-v^2) \, {\rm G_F}^2 c}{\pi \, E} \tag{25}$$

For the crack radius (c), it was decided to use one-half the measured cleavage facet size for test temperatures above  $T_d$  and one-half the measured intergranular facet size for test temperatures of  $T_d$  and below.

As shown in Table 18, the  $\gamma_p^i$  values are in the range of 1.0 x 10<sup>4</sup> to 12.3 x 10<sup>4</sup> ergs/cm<sup>2</sup>. Starting at -196°C for Mo-E2 and Mo-E3 and at -100°C for Mo-E4,  $\gamma_p$  first decreases with increase in temperature up to  $T_d$ , then increases from  $T_d$  to  $T_m$ , and finally decreases above  $T_m$ . This variation is similar to that of  $0_F$  with temperature. The ratio of  $\gamma_p^b$  to  $\gamma_i^a$  at the same temperature varies from 1 to 43. This indicates the possibility that  $\gamma_p^a$  may be larger than  $\gamma_i^a$  even though fracture is controlled by crack initiation.

According to Barrett (15) crack initiation involves the growth of a critical size crack until it encounters a major barrier (typically a grain boundary). In order for the crack to cross the boundary and continue to propagate, an increase in effective surface energy is involved. Thus, it seems reasonable to expect that  $\gamma_p^i$  is generally greater than  $\gamma_i^i$ . For fracture to be controlled by crack initiation rather than crack propagation, the condition to be satisfied is  $(\sigma_F)_p \leq (\sigma_F)_i$ . Based on the results given in Table 17, it appears possible for the ratio of  $(\sigma_F)_p$  to  $(\sigma_F)_i$  to be as low as 1/43 with  $\gamma_p^i \geq \gamma_i^i$ .

#### 5.2 Based on Precracked Charpy Slow Bend Tests

As was shown in Table 10, the fracture toughness values of Mo-E3 as determined by precracked Charpy slow bend tests fall in the range of  $0.4 \times 10^7$  to  $2.1 \times 10^7$  ergs/cm<sup>2</sup>. These values are approximately  $10^3$  times larger than either the calculated effective surface energy for crack initiation ( $\gamma_1^1$ ) or for crack interpolation ( $\gamma_2^1$ ) based on the tensile test results for Mo-E3 as given in the less 17 and 18 respectively. This difference may be due to a greater and of plastic deformation at

Table 18

Calculated Effective Surface Energy for Crack Propagation in Mo-E2, Mo-E3, and Mo-E4 Strip

Material and Original Grain Size	Test Temp.	Fracture Strain ( <sup>E</sup> F)	Elastic Modulus (E) dynes/cm <sup>2</sup>	Facet Semi- Size(c) cm	Fracture Stress $(\frac{\mathbf{F}}{\mathbf{F}})$	Effective Surface Energy(7!) ergs/cm <sup>2</sup>	Fracture Initiation Mode	$\frac{\gamma_{\rm p}'/\gamma_{\rm i}}{\gamma_{\rm p}}$
Mo-E2 (0.026mm)	-196 -80 -55 -30	0.02 0.04 0.35 0.80	3.65×10 <sup>12</sup> 3.3×10 <sup>12</sup> 3.3×10 <sup>12</sup> 3.25×10 <sup>12</sup> 3.25×10 <sup>12</sup>	0.0011 0.0010 0.0010 0.0008	12.3×10 <sup>9</sup> 8.6×10 <sup>9</sup> 10.0×10 <sup>9</sup> 11.6×10 <sup>9</sup> 8.4×10 <sup>9</sup>	5.3x104 2.6x104 3.5x104 3.8x104 2.0x104	intergran. intergran. cleavage cleavage	- 44 112 115 110
Mo-E2 (0.04·tmm)	-196 -75 -50 -25 +25	0.02 0.07 0.20 0.65	3.6×10 <sup>12</sup> 3.3×10 <sup>12</sup> 3.3×10 <sup>12</sup> 3.2×10 <sup>12</sup> 3.2×10 <sup>12</sup>	0.0019 0.0018 0.0020 0.0018	10.9×10 <sup>9</sup> 7.6×10 <sup>9</sup> 8.8×10 <sup>9</sup> 10.1×10 <sup>9</sup> 9.6×10 <sup>9</sup>	7.2x10 <sup>4</sup> 3.6x10 <sup>4</sup> 5.4x10 <sup>4</sup> 6.6x10 <sup>4</sup> 3.0x10 <sup>4</sup>	intergran. intergran. cleavage cleavage	- 14 22 26 12
Mo-E2 (0.174mm)	-196 -40 -20 0 +25	0.02 0.17 0.62 0.95 1.00	3.65×10 <sup>12</sup> 3.3×10 <sup>12</sup> 3.25×10 <sup>12</sup> 3.2×10 <sup>12</sup> 3.2×10 <sup>12</sup>	0.0013 0.0029 0.0034 0.0021	10.3×10 <sup>9</sup> 8.4×10 <sup>9</sup> 9.7×10 <sup>9</sup> 11.0×10 <sup>9</sup> 10.5×10 <sup>9</sup>	4.3×10 <sup>4</sup> 7.1×10 <sup>4</sup> 11.3×10 <sup>4</sup> 9.1×10 <sup>4</sup> 1.0×10 <sup>4</sup>	intergran. intergran. cleavage cleavage	1 18 28 23 20

Table 18 (Continued)

Calculated Effective Surface Energy for Crack Propagation in Mo-E2, Mo-E3, and Mo-E4 Strip

Material		Fracture	e 51254:	į.	Fracture	Effective	1	
Original Grain Size	Test Temp.	Strain $(\epsilon_F)$	Modulus (E)	r acet Semi- Size(c)		Surface Energy( $\gamma_{\rm p}$ )	Fracture Initiation Mode	γ, /γ;
	ပ		dynes/cm <sup>2</sup>		dynes/cm <sup>2</sup>	dynes/cm <sup>2</sup> ergs/cm <sup>2</sup>		1
Mo-E3	961-	0.02	3.65×10 <sup>12</sup>	0.0009	14.8×109	4.2×10 <sup>4</sup>	intergran,	,
(0.023mm)	-85	0.03	3.3×10 <sup>12</sup>	900000	6.1×10 <sup>9</sup>	1.7×10 <sup>4</sup>	intergran.	9
	-45	0.54	3.25×10 <sup>12</sup>	0.0008	11.2×109	$3.6 \times 10^4$	cleavage	36
	-20	0.91	3.25×10 <sup>12</sup>	0,0008	12.4×109	4.3×10 <sup>4</sup>	cleavage	43
	+25	1.06	3.2×10 <sup>12</sup>	0.0008	10.7×10 <sup>9</sup>	3.3×10 <sup>4</sup>	cleavage	33
,			12		ō	4		
ivio - E-4	-100	0.00	3.35×10 <sup></sup>	0.0012	8.6×10′	3.1×10 <sup>±</sup>	intergran.	ı
(0.020mm)	-35	00.0	3.25×10 <sup>12</sup>	0.0009	5.5×109	$1.0 \times 10^4$	intergran.	7
	-25	0.10	$3.2 \times 10^{12}$	0.0009	6.2×109	$1.2 \times 10^4$	intergran.	5
	0	0.36	3.2×10 <sup>12</sup>	0.0010	$7.6 \times 10^{9}$	$2.1 \times 10^{4}$	intergran.	ı۷
	+25	0.68	$3.2 \times 10^{12}$	0.0011	$9.0 \times 10^{9}$	3.2×10 <sup>4</sup>	cleavage	S
	+50	0.61	$3.2 \times 10^{12}$	0.0011	7.5×109	$2.2 \times 10^4$	cleavage	- 7

the tip of the fatigue precrack in the Charpy specimen as compared to the tip of the crack that initiates in a tensile specimen. In other words, the fatigue action at +25°C may produce a relatively "blunt" crack tip corresponding to much lower stress concentration as compared to that for the tip of a tensile test crack formed at -100°C.

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# 6. Variation of Fracture Stress with Temperature

### 6.1 Decrease in Fracture Stress up to Td

Starting from a relatively low temperature,  $-196^{\circ}$ C, the fracture stress ( $\sigma_F$ ) of the various recrystallized molybdenum materials studied in this investigation was found to decrease with increase in test temperature and reach a minimum value at the tensile ductility transition temperature ( $T_d$ ). Since  $\sigma_F$  is slightly higher than the proportional limit over most of this temperature range, it seems likely that a)  $\sigma_F$  corresponds to crack nucleation rather than to crack propagation, and b) crack nucleation requires the prior occurrence of at least a small amount of plastic deformation. A crack that initiates at a stress level higher than that required for propagation would be expected to immediately propagate completely through the specimen section. On this basis, the observed decrease in  $\sigma_F$  up to  $T_d$  is considered to be essentially a consequence of the decrease in yield stress ( $\sigma_V$ ) which occurs with increase in test temperature.

# 6.2 Increase in Fracture Stress Td to Tm

In the range from  $T_d$  to  $T_m$ ,  $\sigma_F$  increases from a minimum to a maximum even though  $\sigma_V$  continues to decrease. These changes are accompanied by an increase in ductility from a relatively low level at  $T_d$  to a relatively high level at  $T_m$ . It was observed that below  $T_d$  the elongation takes place uniformly over the entire gage length, whereas above  $T_d$  necking occurs prior to fracture. These fracture and necking observations indicate that  $T_d$  represents the intersection of the fracture stress corresponding to uniform elongation (i.e., the  $\sigma_F$  curve below  $T_d$ ) with the necking stress ( $\sigma_n$ ) which is the tensile strength on a true stress basis.

As an aid in understanding the increase in  $\mathfrak{I}_F$  in the range from  $T_d$  to  $T_m$ ,  $\mathfrak{I}_F$  can be considered as a flow stress, i.e., the particular flow stress that corresponds to the fracture strain  $(\epsilon_F)$  at a given test temperature. On this basis, it was shown that the increase in  $\mathfrak{I}_F$  above  $T_d$  can be attributed to the following effects: a) strain hardening due to increased substructure formation, b) plastic constraint due to the necking that occurs above  $T_d$ , and c) increase in strain rate due to the localized nature of necking. This approach was first used by Bechtold (8) and more recently by Ault (11), although the latter neglected the strain hardening effect.

For a more complete understanding of the increase in  $\sigma_F$  from  $T_d$  to  $T_m$ , an explanation is needed for the increase in fracture stress per se which permits the flow stress corresponding to  $\epsilon_F$  to increase in the observed manner. Although the plastic constraint and increased local

strain rate effects due to necking both increase the flow stress corresponding to a given amount of plastic strain, it seems doubtful whether these effects also raise the fracture stress per se. It is generally found that an increase in plastic constraint results in a decrease in fracture stress, which is attributed to an increase in the flow stress relative to the fracture stress such that fracture occurs at a smaller plastic strain value than without plastic constraint. Likewise, an increase in strain rate raises the applied stress required for a given amount of plastic strain, and appears to act similar to plastic constraint in that the fracture stress is lowered.

Since no micro-cracks were observed above  $T_d$ , it appears likely that crack initiation rather than crack propagation is the controlling step in the fracture of the molybdenum materials studied. In order to explain the increase in  $0_F$  in the range of  $T_d$  to  $T_m$  on the basis of crack initiation, one might hypothesize that an increase occurs in the effective surface energy for crack initiation  $(\gamma_i)$ . However, calculations of  $\gamma_i$  made on the basis of a modified Cottrell relation using the flow locking parameter  $(k_f)$  instead of the yield locking parameter  $(k_y)$  indicate that, with the possible exception of the Mo-E4 material, there is no significant change in  $\gamma_i$  in this range. Instead, the increase in  $0_F$  appears to be associated in most cases with a decrease in both  $k_f$  and the effective grain size ( $\ell$ ) corrected for the reduction in area that occurs prior to fracture. The decrease in  $k_f$  with increase in temperature can be attributed to decreased association of interstitials with dislocations.

# 6.3 Decrease in Fracture Stress above Tm

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It was found that  ${}^0{}_F$  reaches a maximum at  $T_m$  and then decreases above this temperature. Since no evidence of fibrous fracture was found at the highest test temperature employed (+25°C or +50°C), it does not seem likely that the decrease in  ${}^0{}_F$  can be associated with a change in fracture mode from cleavage to fibrous. However, a change does take place from comparatively undistorted to highly distorted cleavage facets. This is accompanied by an increase in  $k_f$ , which based on the Cottrell relation for crack initiation should tend to lower  ${}^0{}_F$ . Further work is necessary to account for the increase in  $k_f$  above  $T_m$ .

#### IV. CONCLUSIONS

- 1. As determined by tensile tests, the ductility transition temperature  $(T_d)$  and the brittleness transition temperature  $(T_b)$  approximately coincide in fine grain, recrystallized molybdenum. With increase in grain size,  $T_b$  is lowered whereas  $T_d$  is raised.
- 2. The occurrence of a minimum in the observed fracture stress  $(\sigma_F)$  at  $T_d$  is associated with the intersection of the necking stress  $(\sigma_n)$  and the fracture stress corresponding to uniform fracture strain, i.e. necking occurs above  $T_d$  but not below  $T_d$ . If  $T_d$  and  $T_b$  happen to coincide, the yield stress  $(\sigma_y)$  also intersects at the minimum in  $\sigma_F$  since  $T_b$  is defined as the intersection of  $\sigma_F$  and  $\sigma_V$ .
- 3. Taking into account strain hardening due to substructural changes in addition to necking effects such as plastic constraint and increased strain rate, the predicted variation of OF with test temperature above T2 was found to agree (within 15%) with the observed values in most cases.
- 4. For the molybdenum materials with relatively low oxygen contents, the fracture initiation mode is intergranular at or below  $T_d$  and cleavage above  $T_d$ . For a relatively high oxygen content (145 ppm), the fracture initiation mode is intergranular up to a temperature that is intermediate between  $T_d$  and that corresponding to the maximum in  $0_F$   $(T_m)$ .
- 5. By prestraining at a temperature above  $T_d$  and breaking below  $T_d$ , it is possible to increase  $\sigma_F$  by as much as 20% as compared to without prestraining. The increase in  $\sigma_F$  due to prestraining correlates with an increase in cleavage facets in most cases.
- 6. For test temperature below  $T_d$ , fracture occurs after a relatively small amount of plastic strain and is apparently controlled by crack initiation. For test temperatures above  $T_d$ , crack initiation also appears to be the controlling step even though the amount of plastic strain prior to fracture increases markedly.
- 7. The ratio of the flow locking parameter  $(k_f)$  to the yield locking parameter  $(k_y)$  is approximately unity at room temperature and decreases to at least 0.3 with lowering of temperature. A minimum in  $k_f$  was found to occur between  $T_d$  and  $T_m$ .
- 8. Based on the Cottrell relation for crack initiation, the increase in  $\sigma_F$  from  $T_d$  to  $T_m$  is attributed to decreases in both  $k_f$  and the effective grain size. The maximum in  $\sigma_F$  at  $T_m$  is associated with the increase in  $k_f$  after passing through a minimum below  $T_m$ .
- 9. The calculated upper limit of the probable value of the effective surface energy for crack propagation  $(\gamma_p^i)$  was found to be up to about 40 times greater than the corresponding  $\gamma_i^i$  value.

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### Appendix I

# Definitions of Transition Temperatures Used in Text

- $T_b$ .... Brittleness transition temperature corresponds to intersection of the fracture stress ( $\sigma_F$ ) and yield stress ( $\sigma_y$ ) curves.
- $T_{d}$ .... Ductility transition temperature corresponds to minimum in the fracture stress ( $\sigma_F$ ) curve.
- $T_{m}$ .... Corresponds to maximum in fracture stress ( $O_F$ ) curve, which occurs at a temperature above  $T_{d}$ .